QUALITY ASSURANCE PROJECT PLAN
for
SCRAP METAL REMOVAL AND SOIL SAMPLING
at
SCRAP YARDS AND REGULATOR PROCESSING FACILITIES USED BY NICOR GAS

Prepared by
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1. PROJECT DESCRIPTION

1.1 Introduction

This QAPP presents the organization objectives, planned activities, and specific Quality Assurance/Quality Control (QA/QC) procedures associated with the work to be conducted at scrap yards and regulator processing facilities in Illinois. The work is being conducted by Nicor Gas in response to a "CERCLA 106a Letter" dated September 6, 2000.

Specific protocols for sampling, sample handling and storage, chain-of-custody, and laboratory and field analyses will be described. All QA/QC procedures will be structured in accordance with applicable technical standards, U.S. EPA requirements, regulations, guidance, and technical standards. This QAPP has been prepared in general accordance with the U.S. EPA Region 5 QAPP policy, as presented in *U.S. EPA RCRA QAPP Instructions*, and other relevant guidance documents.

This QAPP has been prepared on behalf of Nicor Gas by Huff & Huff, Inc. A *Work Plan* and a *Health and Safety Plan* also have been prepared. All plans are dated September 2000.

1.2 Overall Project Objectives and Decision Statements

One purpose of the proposed work is to gather sufficient information to quantify risk to human health in the event that environmental contamination is determined to be present. The objectives of the proposed work are to determine the nature and extent of potential mercury contamination at the facility.

Overall objectives of the data collection will be as follows:

- Verify and define the nature and extent of mercury on scrap metal and in soil. Data quality must be sufficient to allow comparison with established action levels or regulatory standards (screening levels).

The Decision Statement for this investigation is as follows: What is the nature, risk, and extent of mercury on scrap metal and in soil that presents unacceptable risks, which would therefore warrant remedial action?

Associated specific objectives for field and laboratory data collection are tabulated in Table 1-1.

1.3 Project History

On September 6, 2000, Nicor Gas received a "CERCLA 106a Letter" from the U.S. EPA regarding management of mercury contaminated natural gas regulators at scrap yards and regulator processing facilities in Illinois.

1.4 Proposed Work Activities

The following work will be performed at each site:

(1) Removal of Scrap Metal Piles.
Scrap metal piles containing regulators will be transferred into roll off boxes.

(2) Mercury Management at Mercury Waste Solutions.
The roll off boxes will be transferred to Mercury Waste Solutions in Union Grove, Wisconsin, for mercury management. At Mercury Waste Solutions, all material will be placed on a "tray" and sorted in a negative pressure area (for containment). Material with visible mercury will be sent for retort to reclaim mercury. Material with no visible mercury will be screened for mercury with a Jerome Meter. If the meter reading is above 0.025 mg/m^3 (the NIOSH TWA for elemental mercury), the material will be landfilled as hazardous waste. If the reading is less than 0.025 mg/m^3, the material will be managed as scrap metal.

(3) Soil Evaluation
Once the scrap piles are removed from the site, surficial soil mercury readings will be screened with a Jerome Meter. Readings will be taken in a 10 foot x 10 foot grid. The meter probe will be placed one inch from the ground surface (plus or minus one-half inch). All locations having a reading greater than 0.010 mg/m^3 will be excavated six inches (and landfilled as waste) and the ground surface will be screened again. Once all readings are below 0.010 mg/m^3, soil samples will be collected for laboratory analysis of mercury levels.

TABLE 1-1
EVALUATION PARAMETERS

Constituent	Decision Level a/	Detection Limit	Matrix
Mercury	0.01 mg/m3	0.003 mg/m3	air
Mercury	0.01 mg/kg	0.04 mg/kg	soil

a/ Most stringent decision level. For soil, actual decision level depends in part upon soil pH.

2. PROJECT ORGANIZATION AND RESPONSIBILITY

2.1 Management Responsibilities

U.S. EPA Project Manger/Illinois EPA Project Manager

The U.S. EPA will have the overall responsibility for all phases of the investigation.

Mr. Brad Stimple is the U.S. EPA Project Manager.

Nicor Gas Project Manager

The Nicor Gas Project Manager is responsible for implementing the project and has the authority to commit the resources necessary to meet project objectives and requirements. The Nicor Gas Project Manager's primary function is to ensure that technical, financial, and scheduling objectives are achieved successfully. The Nicor Gas Project Manager will report directly to the U.S. EPA and Illinois EPA Project Managers and will provide the major point of contact and control for matters concerning the project.

Ms. Claudia Macholz is the Nicor Gas Project Manager. (630) 983-8676 Ext. 2456

Huff & Huff Project Manager

The Huff & Huff Project Manager has responsibility for ensuring that the project meets U.S. EPA's objectives and quality standards. The Huff & Huff Project Manager will provide assistance to the Nicor Gas Project Manager in terms of writing and distributing the QAPP to all those parties connected with the project (including the laboratory). The Huff & Huff Project Manager will report directly to the Nicor Gas Project Manager and is responsible for technical QC and project oversight.

Mr. James E. Huff, P.E. is the Huff & Huff Project Manager. (708) 579-5940

2.2 Quality Assurance Responsibilities

Nicor Gas QA Manager

The Nicor Gas QA Manager will have direct access to corporate staff as necessary to resolve any QA dispute. She is responsible for auditing the implementation of the QA program in conformance with the demands of specific investigations, Huff & Huff's policies, and U.S. EPA requirements. The Nicor Gas QA Manager has sufficient authority to stop work on the investigations as deemed necessary in the event of serious QA/QC issues.

Ms. Claudia Macholz is the Nicor Gas QA Manager. (630) 983-8676 Ext. 2456

Huff & Huff QA Manager

The Huff & Huff QA Manager reports directly to the Huff & Huff Project Manager and will be responsible for ensuring that all Huff & Huff procedures for this project are being followed.

Ms. Sarah Monette, P.E. is the Huff & Huff QA Manager. (708) 579-5940

2.3 Laboratory Responsibilities

The laboratory tasked with responsibility for analytical work is Test America, Bartlett, Illinois. (630) 789-3100

Test America Project Manager

The Test America Project Manager will report directly to the Huff & Huff Project Manager and will be responsible for the following:

- Ensuring all resources of the laboratory are available on an as-required basis.
- Overseeing production and final review of analytical reports.

Test America Operations Manager

The Test America Operations Manager will report to the Test America Project Manager and will be responsible for:

- Coordinating laboratory analyses.
- Supervising in-house chain-of-custody.
- Scheduling sample analyses.
- Overseeing data review.
- Overseeing preparation of analytical reports.
- Approving final analytical reports prior to submission to Huff & Huff/Nicor Gas.

Test America Quality Assurance Officer

The Test America QA Officer has the overall responsibility for data after it leaves the laboratory. The Test America QA Officer will communicate data issues through the Test America Project Manager. In addition, the Test America QA Officer will:

- Oversee laboratory QA.
- Oversee QA/QC documentation.
- Conduct detailed data review.
- Determine whether to implement laboratory corrective actions, if required.
- Define appropriate laboratory QA procedures.
- Prepare laboratory SOPs.

Test America Sample Custodian

The Test America Sample Custodian will report to the Test America Operations Manager. Responsibilities of the Test America Sample Custodian will include:

- Receiving and inspecting the incoming sample containers.
- Recording the condition of the incoming sample containers.
- Signing appropriate documents.
- Verifying chain-of-custody.
- Notifying Laboratory Manager and Laboratory Supervisor of sample receipt and inspection.
- Assigning a unique identification number and customer number, and entering each into the sample receiving log.
- With the help of the Laboratory Manager, initiating transfer of the samples to appropriate lab sections.
- Controlling and monitoring access/storage of samples and extracts.

Test America Technical Staff

The Test America Technical Staff will be responsible for sample analysis and identification of corrective actions. The Staff will report directly to the Test America Operations Manager.

2.4 Field Responsibilities

Huff & Huff Field Leader

The Nicor Gas Project Manager will be supported by the Huff & Huff Field Leader. He is responsible for leading and coordinating the day-to-day activities of the various resource specialists under his supervision. The Huff & Huff Field Leader is a highly experienced environmental professional and will report directly to the Nicor Gas Project Manager.

Mr. James E. Huff, P.E. is the Huff & Huff Field Leader. (708) 579-5940

Huff & Huff Field Technical Staff

The Technical Staff for this project will be drawn from Huff & Huff pool of corporate resources. The Technical Staff will be utilized to gather and analyze data, and to prepare various task reports and support materials. All of the designated Technical Staff are experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work.

3. QUALITY ASSURANCE OBJECTIVES

3.1 Precision

3.1.1 Definition

Precision is a measure of the degree to which two or more measurements are in agreement.

3.1.2 Field Precision Objectives

Field precision is assessed through the collection and measurements of field duplicates at a rate of 1 duplicate per 10 analytical samples. Protocols for field duplicate collection are specified in the *Work Plan* (Section 3.2).

3.1.3 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of relative percent differences (RPD) and relative standard deviations (RSD) for three or more replicate samples. The equations to be used for precision in this project can be found in Section 11.1. Precision control limits are given in the applicable SOPs as referenced in Section 7.2.

3.2 Accuracy

3.2.1 Definition

Accuracy is the degree of agreement between an observed value and an accepted reference or true value.

3.2.2 Field Accuracy Objectives

Accuracy in the field is assessed through the use of field and trip blanks and through the adherence to all sample handling, preservation and holding times. Protocols for field and trip blank collection and sample handling procedures are given in the *Work Plan* (Section 3.2).

3.2.3 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the analysis of standard reference materials (SRM), laboratory control samples (LCS) surrogate compounds, and the determination of percent recoveries. The equation to be used for accuracy in this project can be found in Section 11.2. Accuracy control limits are given in the applicable SOPs as referenced in Section 7.2.

3.3 Completeness

3.3.1 Definition

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

3.3.2 Field Completeness Objectives

Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is presented in Section 11.3. The field completeness objective for this project will be greater than 90 percent.

3.3.3 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is presented in Section 11.3. The laboratory completeness objective for this project will be greater than 95 percent.

3.4 Representativeness

3.4.1 Definition

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition within a defined spatial and/or temporal boundary.

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the *Work Plan* is followed and that proper sampling techniques are used. In designing the sampling program, media of concern have been specified.

3.4.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the proper analytical procedures, appropriate methods, meeting sample holding times, and analyzing and assessing field duplicate samples. The sampling network was designed to provide data representative of facility conditions.

3.5 Decision Rules

3.5.1 Definition

A "decision rule" is a statement which allows for a course of action or non-action to be taken, based on assumptions made to draw out and test its logical or empirical consequences.

3.5.2 Decision Rule Objectives

The decision rule objectives address the following.

- Define statistical parameter(s) characterizing the population (e.g., mean, maximum, percentile) and incorporate the scale of decision-making (e.g., residential lot size).
- Identify action level(s).
- Develop "if/then" statements defining conditions that would cause the decision maker to choose among alternative actions (e.g., remediation or no remediation).

Decision rules and levels are specified in Section 1.4 and Table 1-1.

3.6 Comparability

3.6.1 Definition

Comparability is an expression of the confidence with which one data set can be compared to another.

3.6.2 Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the *Work Plan* is followed and that proper sampling techniques are used.

3.6.3 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented in the QAPP. Comparability is also dependent on similar QA objectives.

3.7 Level of Quality Control Effort

Field blank, trip blank, method blank, field duplicate, laboratory duplicate, laboratory control, and standard reference materials (SRM) samples will be analyzed to asses the quality of the data resulting from the field sampling and analytical programs.

- Field and trip blanks consisting of distilled water will be submitted to the analytical laboratories to provide the means to assess the quality of the data resulting from the field sampling program.
- Field blank samples will be analyzed to check for procedural contamination that may cause sample contamination.

- Trip blanks will be used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage.

- Method blank samples will be generated within the laboratory and will be used to assess contamination resulting from laboratory procedures.

- Duplicate samples will be analyzed to check for sampling and analytical reproducibility.

The general level of the QC effort will be one field duplicate and one field blank for every 10 or fewer investigative samples.

The number of duplicate and field blank samples to be collected are specified in the *Work Plan* (Section 3.2).

4. SAMPLING PROCEDURES

The sampling procedures to be used in this site investigation will be consistent for the objectives of this project. The *Work Plan* provides the SOPs for all sampling activities to be conducted during this investigation. The SOPs are presented in the *Work Plan* (Sections 2 and 3).

5. CUSTODY PROCEDURES

5.1 Custody Overview

Custody is one of several factors which are necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files.

A sample or evidence file is under a person's custody if:

- the item is in actual possession of the person.
- the item is in the view of the person after being in actual possession of the person.
- the item was in actual physical possession but is locked up to prevent tampering.
- the item is in a designated and identified secure area.

5.2 Field Custody Procedures

Field logbooks will provide the means of recording data collecting activities performed during the investigation. As such, entries will be described in as much detail as possible so that persons going to the facility could reconstruct a particular situation without reliance on memory.

Field log books will be bound field survey books or notebooks. The title page of each logbook will contain the following:

- Person to whom the logbook is assigned.
- Project name.
- Project start date.
- Project end date.

Entries into the logbook will contain a variety of information. Each entry will include the date, start time, weather, names of all sampling team members present, level of personal protection equipment being used, and the signature of the person making the entry. The names of visitors to the site, field sampling or investigation team personnel and the purpose of their visit also will be recorded in the field logbook.

Measurements made and samples collected will be recorded. All entries will be made in permanent ink, signed, and dated, and no erasures will be made. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the station shall be recorded. All equipment used to make measurements will be identified, along with calibration information.

Samples will be collected following the sampling procedures documented in Section 4. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, and volume and number of containers. Sample identification numbers will be assigned prior to sample collection.

The sample packaging and shipment procedures summarized below will ensure that the samples will arrive at the laboratory with the chain-of-custody intact. An example of a field custody documents is presented in Appendix A.

- The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. Field procedures have been designed such that as few people as possible will handle the samples.
- All bottles will be identified by the use of sample tags with sample numbers, sampling locations, date/time of collection, and type of analysis.
- Samples will be accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record.
- Samples will be properly packaged on ice at 4 C for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be secured with strapping tape for shipment to the laboratory.

5.3 Laboratory Custody Procedures

Laboratory custody procedures for sample receiving and log-in; sample storage and numbering; tracking during sample preparation and analysis; and storage of data are described in a laboratory SOP provided in Appendix B.

5.4 Final Evidence Files

The final evidence file will be the central repository for all documents which constitute evidence relevant to sampling and analysis activities as described in this QAPP. Huff & Huff is the custodian of the evidence file and maintains the contents of evidence files for the investigation, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and data reviews in a secured, limited access area.

The final evidence file will include at a minimum:

- Field logbooks.
- Field data and data deliverables.
- Photographs.
- Drawings.
- Soil boring logs.
- Laboratory data deliverables.
- Progress reports, QA reports, interim project reports, etc.
- All custody documentation (tags, forms, etc.)

6. CALIBRATION PROCEDURES AND FREQUENCY

6.1 Field Instrument Calibration

The Jerome Meter will be the only field instrument used. The field instrument requires annual calibration at the factory as described in Appendix C.

6.2 Laboratory Instrument Calibration

Calibration procedures for a specific laboratory instrument will consist of initial calibrations (3 or 5-points), initial calibration verifications, and continuing calibration verification. For a description of the calibration procedures for a specific laboratory instrument, refer to the applicable SOPs in Appendix B. The SOP for each analysis performed in the laboratory describes the calibration procedures, their frequency, acceptance criteria and the conditions that will require recalibration. In all cases, the initial calibration will be verified using an independently prepared calibration verification solution.

The laboratory maintains a sample logbook for each instrument which will contain the following information: instrument identification, serial number, date of calibration, analyst, calibration solutions run, and the samples associated with these calibrations.

7. ANALYTICAL PROCEDURES

7.1 Field Analytical Procedures

The procedures for field analytical determinations using the Jerome Meter are described in Section 1.4. The standardization and QA criteria for these parameters are provided in Appendix C.

7.2 Laboratory Analytical Procedures

Test America laboratory will implement the project required SOPs. These laboratory SOPs for sample preparation, cleanup, and analysis are based on SW-846. These SOPs provide sufficient details and are specific to this investigation.

The documentation of appropriate method validation is submitted in Appendix B. It includes the criteria for acceptance, rejection or qualification of data.

TABLE 7-1
SUMMARY OF ANALYTICAL PROCEDURES

Analyte	Lab. SOP No.	Equivalent EPA Method Number [a/]
Mercury	SOP BT04-04.4	7471-A
pH	SOP "pH" #5	9045-C

[a/] SW-846

7.2.1 List of Project Target Compounds and Laboratory Detection Limits

The target compound is mercury. Refer to Section 1.4 for mercury detection limits.

7.2.2 List of Associated Quality Control Samples

The laboratory SOPs listed in Table 7-1 include a QC section which addresses the minimum QC requirements for the analysis of specific analyte groups.

8. INTERNAL QUALITY CONTROL CHECKS

8.1 Field Quality Control Checks

QC procedures for the Jerome Meter will include calibrations as described in Section 6.1, measuring duplicate samples, and checking the reproducibility of the measurements by taking multiple readings on a single sample or reference standard. Assessment of field sampling precision and bias will be made by collecting field duplicates and field blanks for laboratory analysis. Collection of the samples will be in accordance with the *Work Plan*.

8.2 Laboratory Quality Control Checks

Test America has a QC program in place to ensure the reliability and validity of the analysis performed at the laboratory. All analytical procedures are documented in writing as SOPs and each SOP includes a QC section which addresses the minimum QC requirements for the procedure. The internal QC checks differ slightly for each individual procedure but in general the QC requirements include the following:

- Method blanks
- Reagent/preparation blanks
- Instrument blanks
- Surrogate spikes
- Laboratory duplicates
- Laboratory control standards

All data obtained will be properly recorded. The data package will include a full deliverable package capable of allowing the recipient to reconstruct QC information and compare it to QC criteria. Any samples analyzed in nonconformance with the QC criteria will be reanalyzed by the laboratory, if sufficient volume is available. It is expected that sufficient volumes/weights of samples will be collected to allow for reanalysis when necessary.

9. DATA COLLECTION

9.1 Data Reduction

9.1.1 Field Data Reduction Procedures

All field data will be written into field log books immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. Later, when the results calculation forms required for this study are being filled out, the Field Manager will review the forms to determine whether any errors have been made by the field crew.

9.1.2 Laboratory Data Reduction Procedures

Laboratory data reduction procedures will be performed according to the following protocol. All raw analytical data will be recorded in numerically identified laboratory notebooks. These notebooks will be issued only by the Laboratory QA Manager. Data are recorded in this notebook along with other pertinent information, such as the sample identification number and the sample tag number. Other details will also be recorded in the lab notebook, such as the analytical method used (SOP), name of analyst, the date of analysis, matrix sampled, reagent concentrations, instrument settings, and the raw data. Each page of the notebook shall be signed and dated by the analyst. Copies of any strip chart printouts (such as gas chromatograms) will be maintained on file. Periodic review of these notebooks by the Lab QA Manager takes place prior to final data reporting. (Records of notebook entry inspections are maintained by the Lab QA Manager.)

For this project, the equations that will be employed in reducing data are presented in the associated SOPs. The formulae included in the SOPs make pertinent allowance for matrix type. All calculations are checked by the Laboratory QA Manager at the conclusion of each operating day. Errors are noted, corrections are made, but the original notations are crossed out legibly. Analytical results for soil samples shall be calculated and reported on a dry weight basis.

QC data (e.g. laboratory duplicates, surrogates) will be compared to the method acceptance criteria. Data considered to be acceptable will be entered into the laboratory computer system. Data summaries will be sent to the Laboratory QA Manager for review. If approved, data will be logged into the project database format. Unacceptable data shall be appropriately qualified in the project report. Case narratives will be prepared which will include information concerning data that fell outside acceptance limits, and any other anomalous conditions encountered during sample analysis.

9.2 Data Validation

9.2.1 Procedures Used to Validate Field Data

The procedures to evaluate field data for this investigation include checking for transcription errors and review of field logbooks, on the part of field crew members. This task will be the responsibility of the Field Manager.

9.2.2 Procedures Used to Validate Laboratory Data

One hundred percent of the analytical data shall be validated.

The overall completeness of the data package will be evaluated by a Data Validator. *Completeness checks will be administered on all data to determine whether deliverables* specified in the QAPP are present. At a minimum, deliverables will include chain-of-custody forms, analytical results, and QC summaries. The Data Validator will determine whether all required items are present and request copies of missing deliverables.

9.3 Data Reporting

9.3.1 Field Data Reporting

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of all measurements made in the field, and documentation of all field calibration activities.

9.3.2 Laboratory Data Reporting

The task of reporting laboratory data (to the U.S. EPA) begins after the independent validation activity has been concluded. The Huff & Huff QA Manager must perform a final review of the report summaries and case narratives to determine whether the report meets project requirements.

10. PREVENTATIVE MAINTENANCE

10.1 Field Instrument Preventative Maintenance

The field equipment for this project includes a Jerome Meter. Specific preventative maintenance procedures to be followed for field equipment are based on those recommended by the manufacturer. Field instruments will be checked daily before use.

10.2 Laboratory Instrument Preventative Maintenance

As part of the QA Program Plan, a routine preventative maintenance program is conducted by Test America to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees regularly perform routine scheduled maintenance and repair of [or coordinate with the vendor for the repair of] all instruments. All maintenance that is performed is documented in the laboratory's operating record. All laboratory instruments are maintained in accordance with manufacturer's specifications.

11. SPECIFIC ROUTINE PROCEDURES USED TO EVALUATE DATA PRECISION, ACCURACY, AND COMPLETENESS

11.1 Accuracy Assessment

In order to assure the accuracy of the analytical procedures, an environmental sample shall be spiked with a known amount of mercury. At a minimum, one sample spike should be included in every set of 20 samples tested on each instrument, for each sample matrix to be tested (i.e., soil). The increase in concentration of the analyte observed in the spiked sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample, determines the percent recovery.

Accuracy is similarly assessed by determining percent recoveries for surrogate compounds added to each field and QC sample and will also be further assessed through determination of percent recoveries for laboratory control samples.

Percent recovery for MS/MSD results is determined according to the following equation:

$$\%\ R = \frac{(\text{Amount in Spiked Sample} - \text{Amount in Sample})}{\text{Known amount added}} \times 100$$

Percent recovery for LCS and surrogate compound results is determined according to the following equation:

$$\%\ R = \frac{\text{Experimental Concentration}}{\text{Known amount added}} \times 100$$

11.2 Precision Assessment

The relative percent difference (RPD) between the spike and matrix spike, or matrix spike and sample duplicate in the case of metals, and field duplicate pair or laboratory duplicate pair is calculated to compare to precision DQOs and plotted. The RPD is calculated according to the following formula.

$$RPD = \frac{(\text{Amount in Sample 1} - \text{Amount in Sample 2})}{0.5\ (\text{Amount is Sample 1} + \text{Amount in Sample 2})} \times 100$$

11.3 Completeness Assessment

Completeness is the ratio of the number of valid sample results to the total number of samples analyzed with a specific matrix and/or analysis. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness} = \frac{(\text{number of valid measurements})}{(\text{number of measurements planned})} \times 100$$

11.4 Assessment of Data

The field and laboratory data collected during this investigation will be used to evaluate the nature and extent of contamination at the site. The QC results will be compared to the objectives presented in Section 1.4. Only data generated in association with QC results meeting these objectives will be considered useable for decision making purposes.

In addition, the data obtained will be both qualitatively and quantitatively assessed on a project-wide, matrix-specific, parameter-specific and unit-specific basis. This assessment will be performed by the Huff & Huff QA Manager and the results presented and discussed in detail in the final investigation report. Factors to be considered in this assessment of field and laboratory data will include, but not necessarily be limited to, the following.

- Were all samples obtained using the methodologies and SOPs proposed in the QAPP?
- Were all proposed analyses performed according to the SOPs provided in the QAPP?
- Were samples obtained from all proposed sampling locations and depths?
- Do any analytical results exhibit elevated detection limits due to matrix interferences or contaminants present at high concentrations?
- Were all field and laboratory data validated according to the validation protocols, including project-specific QC objectives, proposed in the QAPP?
- Which data sets were found to be unusable (qualified as "R") based on the data validation results?
- Which data sets were found to be usable for limited purposes (qualified as "J") based on the data validation results?
- What affect do qualifiers applied as a result of data validation have on the ability to implement the project decision rules?
- Were the project-specific decision rules used as proposed?
- For any cases where the proposed procedures and/or requirements have not been met, has the effect of these issues on the project objectives been evaluated?
- Have any remaining data gaps been identified and summarized in the final investigation report?
- Based on the overall findings of the investigation and this assessment, were the original project objectives appropriately defined? If not, have revised project objectives been developed?

APPENDIX A

TESTAMERICA INC.

Chain of Custody Record

Page____ of ____

❒ Asheville, NC (A) (828) 254 5169	❒ Bartlett, IL (C) (630) 289-3100	❒ Cedar Falls, IA (E) (319) 277-2401	❒ Charlotte, NC (G) (704) 392-1164	❒ Dayton, OH (I) (937) 294-6856	❒ Lumberton, NC (K) (910) 738-6190	❒ Nashville, TN (M) (615) 726-0177	❒ Pontiac, MI (O) (248) 332-1940	❒ Rockford, IL (Q) (815) 874-2171
❒ Atlanta, GA (B) (770) 368-0636	❒ Brighton, CO (D) (303) 659-0497	❒ Charleston, SC (F) (843) 849-6550	❒ Columbia, SC (H) (803) 796-8989	❒ Davenport, IA (J) (319) 323-7944	❒ Indianapolis, IN (L) (317) 842-4261	❒ Macon, GA (N) (912) 757-0811	❒ Orlando, FL (P) (407) 851-2560	❒ Watertown, WI (R) (920) 261-1660

Client:	Project No.:
Report Address:	Invoice Address:
Attn:	Attn:
Phone No.:	Sampled By:
Fax No.:	P.O. No:
TURNAROUND TIME	Quote No.
❒ Standard	State Samples Collected
❒ Rush (surcharges may apply)	Date Needed: _____

REQUESTED PARAMETERS

Is this work being conducted for regulatory compliance monitoring? Yes___ No___

Is this work being conducted for regulatory enforcement action? Yes___ No___

Which regulations apply:
RCRA____ NPDESWastewater____
UST____ Drinking Water____
Other___ None____

Sample ID	Date	Time	Comp (C) Grab (G)	Matrix	Lab Use	# and type of containers: HCl	NaOH	HNO_3	H_2SO_4	Other	None	REMARKS

QC Deliverables: ❒ None ❒ Level 2 - Batch QC ❒ Level 3 ❒ Level 4 ❒ Other

Init Lab Temp **Rec Lab Temp**

COMMENTS:

Relinquished By:	Date / Time	Received By:	Date / Time	**LAB USE ONLY:**
Relinquished By:	Date / Time	Received By:	Date / Time	
Relinquished By:	Date / Time	Received By:	Date / Time	**Custody Seal:** ❒ Yes ❒ No ❒ N/A

APPENDIX B

STATE OF ILLINOIS

ENVIRONMENTAL PROTECTION AGENCY

ENVIRONMENTAL LABORATORY ACCREDITATION

is hereby granted to

TESTAMERICA INC. BARTLETT DIVISION

850 W. BARTLETT RD.

BARTLETT, IL 60103-4400

ACCREDITATION NUMBER #100221

According to the Illinois Administrative Code, Title 35, Subtitle A, Chapter II, Part 186, ACCREDITATION OF LABORATORIES FOR DRINKING WATER, WASTEWATER AND HAZARDOUS WASTES ANALYSIS, the State of Illinois formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed below.

The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part 186 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part 186. Please contact the Illinois EPA Environmental Laboratory Accreditation Program (IL ELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Illinois is not an endorsement or a guarantee of validity of the data generated by the laboratory.

Jeri Long

Jeri Long
Accreditation Officer
Environmental Laboratory Accreditation Program

Certificate No.: 000142
Expiration Date: 05/22/2001
Issued On: 07/05/2000
Date of Last On-Site Assessment: 3/21/2000 - 3/23/2000

State of Illinois
Environmental Protection Agency
Awards the Certificate of Approval

Certificate No.: 000142

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Drinking Water, Inorganic

SM2320B,18Ed
Alkalinity

SM2510B,18Ed
Conductivity

SM2540C,18Ed
Total dissolved solids

SM2550,18Ed
Temperature

SM3111B,18Ed
Sodium

SM4500F-C,18Ed
Fluoride

SM4500NO2B,18Ed
Nitrite

SM4500NO3D,18Ed
Nitrate

SM4500P-E,18Ed
Orthophosphate

SM5540-C,18Ed
Foaming agent

USEPA150.1
Hydrogen ion (pH)

USEPA200.7R4.4

Aluminum	Barium	Beryllium
Cadmium	Calcium	Chromium
Copper	Iron	Manganese
Nickel	Silver	Zinc

USEPA200.9R2.2

Arsenic	Lead	Selenium

USEPA245.1R3.0
Mercury

USEPA335.4R1.0
Cyanide

Drinking Water, Organic

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Drinking Water, Organic *USEPA524.2R4.1*

Total trihalomethanes	Vinyl chloride	Volatile organic contaminants regulated
Volatile organic contaminants unregulated		

Hazardous and Solid Waste, Inorganic

1010

Ignitability

1030

Ignitability of Solids

1311

TCLP (Organic and Inorganic)

6010B

Aluminum	Antimony	Arsenic
Barium	Beryllium	Boron
Cadmium	Calcium	Chromium
Cobalt	Copper	Iron
Lead	Magnesium	Manganese
Molybdenum	Nickel	Selenium
Silver	Strontium	Thallium
Tin	Titanium	Vanadium
Zinc		

7060A

Arsenic

7140

Calcium

7421

Lead

7450

Magnesium

7470A

Mercury

7471A

Mercury

7610

Potassium

7740

Selenium

7760A

Silver

7770

Sodium

9012A

Cyanide

9034

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Hazardous and Solid Waste, Inorganic	*9034*	Sulfides
9038		
Sulfate		
9040B		
Hydrogen Ion (pH)		
9045C		
Hydrogen Ion (pH)		
9050A		
Specific Conductance		
9060		
Total Organic Carbon (TOC)		
9066		
Phenolics		
9070		
Total Recoverable Oil & Grease		
9210		
Nitrate		
9214		
Fluoride		
9251		
Chloride		
Hazardous and Solid Waste, Organic		
8081A		
4,4'-DDD	4,4'-DDE	4,4'-DDT
Aldrin	alpha-BHC	alpha-Chlordane
beta-BHC	Chlordane - not otherwise specified	delta-BHC
Dieldrin	Endosulfan I	Endosulfan II
Endosulfan sulfate	Endrin	Endrin aldehyde
Endrin ketone	gamma-BHC (Lindane)	gamma-Chlordane
Heptachlor	Heptachlor epoxide	Methoxychlor
Toxaphene		
8082		
PCB-1016	PCB-1221	PCB-1232
PCB-1242	PCB-1248	PCB-1254
PCB-1260		
8141A		
Atrazine	Chloropyrifos	Diazinon
Dimethoate	Fonophos	Parathion ethyl
Phorate	Simazine	Terbufos
8151A		
2,4,5-T	2,4,5-TP (Silvex)	2,4-D
Dalapon	Dicamba	Dichloroprop
Dinoseb	Pentachlorophenol	Picloram

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Hazardous and Solid Waste, Organic	*8260B*	
1,1,1,2-Tetrachloroethane	1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane	1,1-Dichloroethane	1,1-Dichloroethene
1,1-Dichloropropene	1,2,3-Trichlorobenzene	1,2,3-Trichloropropane
1,2,4-Trichlorobenzene	1,2,4-Trimethylbenzene	1,2-Dibromo-3-chloropropane (DBCP)
1,2-Dibromoethane (EDB)	1,2-Dichlorobenzene	1,2-Dichloroethane
1,2-Dichloropropane	1,3,5-Trimethylbenzene	1,3-Dichlorobenzene
1,3-Dichloropropane	1,4-Dichlorobenzene	1,4-Dioxane
2,2-Dichloropropane	2-Butanone (Methyl ethyl ketone, MEK)	2-Chloro-1,3-butadiene (Chloroprene)
2-Chloroethyl vinyl ether	2-Chlorotoluene	2-Hexanone
2-Nitropropane	4-Chlorotoluene	4-Methyl-2-pentanone (Methyl isobutyl ketone, MI
Acetone	Acetonitrile	Acrolein (Propenal)
Acrylonitrile	Allyl chloride	Benzene
Benzyl chloride	Bromobenzene	Bromochloromethane
Bromodichloromethane	Bromoform	Bromomethane
Carbon disulfide	Carbon tetrachloride	Chlorobenzene
Chlorodibromomethane (Dibromochloromethane)	Chloroethane	Chloroform
Chloromethane	cis-1,2-Dichloroethene	cis-1,3-Dichloropropene
cis-1,4-Dichloro-2-butene	Dibromomethane	Dichlorodifluoromethane
Dichloromethane (Methylene chloride)	Diethyl ether	Ethyl acetate
Ethyl methacrylate	Ethylbenzene	Hexachlorobutadiene
Isopropylbenzene	m-Xylene	Methacrylonitrile
Methyl iodide (Iodmethane)	Methyl methacrylate	Methyl-t-butyl ether
n-Butylbenzene	n-Propylbenzene	Naphthalene
o-Xylene	p-Isopropyltoluene	p-Xylene
Paraldehyde	Pentachloroethane	Propionitrile (Ethyl cyanide)
sec-Butylbenzene	Styrene	tert-Butylbenzene
Tetrachloroethene	Toluene	trans-1,2-Dichloroethene
trans-1,3-Dichloropropene	trans-1,4-Dichloro-2-butene	Trichloroethene
Trichlorofluoromethane	Vinyl acetate	Vinyl chloride
8270C		
1,2,4-Trichlorobenzene	1,2-Dichlorobenzene	1,2-Diphenylhydrazine
1,3-Dichlorobenzene	1,4-Dichlorobenzene	2,4,5-Trichlorophenol
2,4,6-Trichlorophenol	2,4-Dichlorophenol	2,4-Dimethylphenol
2,4-Dinitrophenol	2,4-Dinitrotoluene (2,4-DNT)	2,6-Dichlorophenol
2,6-Dinitrotoluene (2,6-DNT)	2-Chloronaphthalene	2-Chlorophenol
2-Methylnaphthalene	2-Nitroaniline	2-Nitrophenol
3,3'-Dichlorobenzidine	3-Nitroanaline	4,6-Dinitro-2-methylphenol
4-Bromophenyl phenyl ether	4-Chloro-3-methylphenol	4-Chloroanaline
4-Chlorophenyl phenyl ether	4-Nitroanaline	4-Nitrophenol
Acenaphthene	Acenaphthylene	Anthracene
Benzidine	Benzo(a)anthracene	Benzo(a)pyrene
Benzo(b)fluoranthene	Benzo(g,h,i)perlyene	Benzo(k)fluoranthene
Benzoic acid	Benzyl alcohol	Bis(2-chloroethoxy) methane
Bis(2-chloroethyl) ether	Bis(2-chloroisopropyl) ether	Bis(2-ethylhexyl) phthalate
Butyl benzyl phthalate	Chrysene	Di-n-butyl phthalate
Di-n-octyl phthalate	Dibenzo(a,h)anthracene	Dibenzofuran

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Hazardous and Solid Waste, Organic	*8270C*	Diethyl phthalate
Dimethyl phthalate	Fluoranthene	Fluorene
Hexachlorobenzene	Hexachlorobutadiene	Hexachlorocyclopentadiene
Hexachloroethane	Indeno(1,2,3-cd) pyrene	Isophorone
m-Cresol (3-Methylphenol)	N-Nitrosodi-n-propylamine	N-Nitrosodimethylamine
N-Nitrosodiphenylamine	Naphthalene	Nitrobenzene
o-Cresol (2-Methylphenol)	p-Cresol (4-Methylphenol)	Pentachlorophenol
Phenanthrene	Phenol	Pyrene
Pyridine		
8310		
Acenaphthene	Acenaphthylene	Anthracene
Benzo(a)anthracene	Benzo(a)pyrene	Benzo(b)fluoranthene
Benzo(g,h,i)perylene	Benzo(k)fluoranthene	Chrysene
Dibenzo(a,h)anthracene	Fluorene	Fluroanthene
Indeno(1,2,3-cd) pyrene	Naphthalene	Phenanthrene
Pyrene		
Wastewater, Inorganic		
HACH8000		
Chemical Oxygen Demand (COD)		
SM2320B,18Ed		
Alkalinity		
SM2510B,18Ed		
Specific Conductance		
SM3111B,18Ed		
Potassium	Sodium	
SM3500Cr-D,18Ed		
Chromium VI		
SM4500CN-CG18Ed		
Cyanide-amenable to chlorination		
SM4500NO2B,18Ed		
Nitrite		
SM4500P-E,18Ed		
Orthophosphate (as P)	Phosphorus	
SM5210B,18Ed		
Biochemical Oxygen Demand (BOD)	Carboneous Biochemical Oxygen Demand (CBOI	
SM5310C,18Ed		
Total organic carbon (TOC)		
SM5520B,18Ed		
Oil and Grease		
SM5540C,18Ed		
Surfactants		
USEPA150.1		
Hydrogen Ion (pH)		

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Wastewater, Inorganic	*USEPA160.1*	
Residue (TDS)		
USEPA160.2		
Residue (TSS)		
USEPA160.3		
Residue (Total)		
USEPA160.4		
Residue (Volatile)		
USEPA160.5		
Residue (Settable solids)		
USEPA200.7		
Aluminum	Antimony	Arsenic
Barium	Beryllium	Boron
Cadmium	Chromium	Cobalt
Copper	Iron	Lead
Magnesium	Manganese	Molybdenum
Nickel	Selenium	Silver
Thallium	Tin	Vanadium
Zinc		
USEPA239.2		
Lead		
USEPA245.1		
Mercury		
USEPA270.2		
Selenium		
USEPA325.2		
Chloride		
USEPA330.5		
Chlorine		
USEPA335.4R1.0		
Cyanide		
USEPA350.1		
Ammonia		
USEPA351.2		
Kjeldahl Nitrogen		
USEPA375.4		
Sulfate		
USEPA420.2		
Phenolics		
Wastewater, Organic		
USEPA608		
4,4'-DDD	4,4'-DDE	4,4'-DDT
Aldrin	alpha-BHC	beta-BHC

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Wastewater, Organic	*USEPA608*	
delta-BHC	Dieldrin	Chlordane
Endosulfan II	Endosulfan sulfate	Endosulfan I
Endrin aldehyde	gamma-BHC (Lindane)	Endrin
Heptachlor epoxide	PCB-1016	Heptachlor
PCB-1232	PCB-1242	PCB-1221
PCB-1254	PCB-1260	PCB-1248
USEPA624		Toxaphene
1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane	1,1,2-Trichloroethane
1,1-Dichloroethane	1,1-Dichloroethene	1,2-Dichlorobenzene
1,2-Dichloroethane	1,2-Dichloropropane	1,3-Dichlorobenzene
1,4-Dichlorobenzene	2-Chloroethylvinyl ether	Acrylonitrile
Benzene	Bromodichloromethane	Bromoform
Bromomethane	Carbon tetrachloride	Chlorobenzene
Chloroethane	Chloroform	Chloromethane
cis-1,3-Dichloropropene	Dibromochloromethane	Dichloromethane (Methylene chloride)
Ethylbenzene	Tetrachloroethene	Toluene
trans-1,2-Dichloroethene	trans-1,3-Dichloropropene	Trichloroethene
Trichlorofluoromethane	Vinyl chloride	
USEPA625		
1,2,4-Trichlorobenzene	1,2-Dichlorobenzene	1,3-Dichlorobenzene
1,4-Dichlorobenzene	2,3-Dinitrophenol	2,4,6-Trichlorophenol
2,4-Dichlorophenol	2,4-Dimethylphenol	2,4-Dinitrophenol
2,4-Dinitrotoluene (2,4-DNT)	2,6-Dinitrotoluene (2,6-DNT)	2-Chloronaphthalene
2-Chlorophenol	2-Methyl-4,6-dinitrophenol	2-Nitrophenol
3,3'-Dichlorobenzidine	4-Bromophenyl phenyl ether	4-Chloro-3-methylphenol
4-Chlorophenyl phenyl ether	4-Nitrophenol	Acenaphthene
Acenaphthylene	Anthracene	Benzidine
Benzo(a)anthracene	Benzo(a)pyrene	Benzo(b)fluoranthene
Benzo(g,h,i)perylene	Benzo(k)fluoranthene	Benzyl butyl phthalate
Bis(2-chloroethoxy) methane	Bis(2-chloroethyl) ether	Bis(2-ethylhexyl) phthalate
Chrysene	Di-n-butyl phthalate	Di-n-octyl phthalate
Dibenzo(a,h)anthracene	Diethyl phthalate	Dimethyl phthalate
Fluorene	Fluroanthene	Hexachlorobenzene
Hexachlorobutadiene	Hexachlorocyclopentadiene	Hexachloroethane
Ideno(1,2,3-cd) pyrene	Isophorone	N-Nitrosodi-n-propylamine
N-Nitrosodimethylamine	N-Nitrosodiphenylamine	Naphthalene
Nitrobenzene	Pentachlorophenol	Phenanthrene
Phenol	Pyrene	

STANDARD OPERATING PROCEDURE

TestAmerica Incorporated
Bartlett, IL Division

Title: **NON-AQUEOUS MERCURY ANALYSIS BY CVAA-FIMS SYSTEM**

SOP No.: BT04-04.4
Revision: 4
Date: March 14, 2000

Computer File Name: C:/SOPS/ METSOP/METALS/BT04-04.4 Hg Non-aqueous 3-14-00

Division Manager Approval	3/20/00 Date	Quality Assurance Approval	3/20/00 Date
Technical Approval	3/20/2000 Date	Operations Manager Approval	3-20-00 Date

This method may involve hazardous materials, operations and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed toe, nonabsorbent shoes are a minimum. For specific hazard(s) see reagents, materials and procedure sections of this SOP.

METHOD REFERENCES: SW-846 Method 7471A

ANALYTE: MERCURY, NON-AQUEOUS

INSTRUMENTATION: CVAA - FIMS

Table of Contents

1.0 SCOPE AND APPLICATION

This method is applicable to the analysis of mercury in non-aqueous matrices utilizing cold vapor atomic absorption (FIMS system). Table 1 covers the analytes and their respective reporting limits (RL). This method is restricted to use by or under the supervision of analysts experienced in the use of the Cold Vapor Atomic Absorption FIMS System. Each analyst must demonstrate the ability to generate acceptable results with this method.

Table 1. Reporting Limits of Analytes Amenable to this SOP

Analyte	Non-aqueous Reporting Limit (mg/Kg)
Mercury	0.04

The reporting limits listed in the SOP are given as guidance only, and are subject to change without notice based upon operating conditions, project specific requirements, and the results of validation studies.

1.1 Definitions.

Analytical Batch - A set of up to 10 samples plus QCIs that are processed in the same instrument on the same calendar day. Routine QC including blanks, MS/MSDs, and CCVS/LCSs are not included in the 10 count. All other QC samples, including those originating from clients and those originating internally (MVSs, MDLs, and PEs), must be included in the 10 count. The lot number of the reagents cannot be changed in the middle of an analytical batch.

Preparation Batch - A set of up to 10 samples of the same matrix that are prepped on the same calendar day by the same analyst using the same techniques. Routine QCIs including blanks, MS/MSDs, and LCS/LCSDs are not included in the 10 count. All other QC samples, including those originating from clients and those originating internally (MVS, MDLs, PEs) must be included in the 10 count. The lot number of the reagents cannot be changed in the middle of a preparation batch.

NIST - The United States Department of Commerce, Technology Administration, National Institute of Standards and Technology (formerly, National Bureau of Standards).

Quality Control Indicators (QCIs) - A general term used to refer to blanks, standards, MS/MSDs, etc.

Dissolved Metals - Those constituents (metals) which will pass through a 0.45 micron membrane filter.

Suspended Metals - Those constituents (metals) which are retained by a 0.45 micron membrane filter.

Total Metals - The concentration of metals in an unfiltered sample following vigorous digestion.

2.0 SUMMARY OF METHOD

Mercury analysis by cold vapor atomic absorption is based upon the absorption of radiation at the 253.7 nm wavelength by mercury vapor.

A known amount of sample is treated with aqua regia and water followed by heating at 95°C for 2 minutes. The sample is then treated with potassium permanganate and water, and oxidized at 95°C for 30 minutes. This digestion procedure converts the mercury to the mercuric ion. Excess permanganate is reduced with the addition of hydroxylamine hydrochloride.

The addition of stannous chloride at the instrument reduces the mercury to the elemental vapor state. The mercury vapor is produced and contained in a closed system in which the mercury vapor passes through a cell positioned in the light path of the atomic absorption spectrometer. The absorption of the mercury vapor from radiation at 253.7 nm is measured and quantitated against the absorption of known mercury standards.

3.0 INTERFERENCES

Organic compounds that have an absorbance at 253.7nm will produce interference. If highly volatile organic compounds are suspected, a preliminary analysis of the digested samples prior to addition of stannous chloride would determine if this type of interference is present.

Sulfide is a known interference which can be eliminated by the careful addition of the potassium permanganate reagent.

Samples high in chlorides, such as industrial effluents and brines, require the addition of excess potassium permanganate. During the digestion step, chlorides can be converted to free chlorine which will absorb at 253.7 nm. This can be avoided by using excess hydroxylamine sulfate reagent and shaking the sample digest container prior to the addition of the stannous chloride reagent.

Copper has been reported to interfere. However, copper concentrations as high as 10 ppm had no effect on recovery of mercury from spiked samples.

Low mercury concentrations (below 10ug/L) may be absorbed on the walls of the sample digest containers, this depends on the material used. The containers are checked for this behavior by analyzing a reporting limit verification standard (RLVS).

4.0 EQUIPMENT AND SUPPLIES

The following apparatus is recommended for performing this procedure. Equivalent items can be used so long as the analytical and QA/QC requirements in this SOP can be met.

4.1 Perkin Elmer FIMS 100.

Analytical system complete with autosampler and data system for [illegible] and analysis.

4.1.1 FIMS Cell.

4.1.2 Data System - The data acquisition system must be capable of time stamping all data produced with the correct date and time.

4.1.3 Argon Gas Supply - Liquid argon, 99.99% pure or better.

4.2 Pipettes - Calibrated eppendorf pipettes with disposable tips.

4.3 Glassware - Beakers, graduated cylinders and volumetric flasks. All glassware should be washed according to the procedures given in the Glassware Washing SOP.

4.4 Water Bath - With covered top and capable of maintaining [illegible] water at 95°C.

4.5 BOD Bottles - Glass BOD bottles, 300mL.

5.0 REAGENTS AND STANDARDS

5.1. Reagents

Reagent grade chemicals (or better) shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.1.1 Traceability. See the Reagent and Standards Tracking SOP for instructions on ensuring traceability.

5.1.2 Metals-free reagent water. All references to reagent water in this SOP refer to metals-free reagent water, defined as water that does not contain interferences at or above the reporting limit.

5.1.3 Aqua Regia. Fisher brand ACS Plus grade or equivalent. Assayed mercury must be less than or equal to 1 ug/L.

5.1.4 Potassium Permanganate Solution (5% w/v). Dissolve 5g of potassium permanganate in 100mL of reagent water. Shelf life: 1 year.

5.1.5 Potassium Permanganate Solution (0.5% w/v). Prepare a 1/10 dilution of the 5% solution prepared in section 5.1.4. Shelf life: Until parent solution expires.

5.1.6 Sodium Chloride Hydroxylamine Hydrochloride Solution. Dissolve 12g of sodium chloride and 12g of hydroxylamine hydrochloride in 100mL reagent water. Hydroxylamine sulfate may be substituted for hydroxylamine hydrochloride. Shelf life: 1 year.

5.1.7 Stannous Chloride (1.1% solution w/v). Add 11g of stannous chloride to 10mL of reagent water. Slowly add 30mL of concentrated hydrochloric acid and then bring up to a final volume of 1L with reagent water. This mixture is a suspension and must be stirred continually on a magnetic stir plate during use. Shelf life: prepare fresh daily.

5.2 Standards

The following standards are recommended for performing this procedure. The use of alternative standards will be allowed as long as the analytical and quality objectives of the SOP can be met. When instructions are given on how to prepare a specific volume of standard, larger or smaller volumes can be prepared as needed. At least one of the standards should be NIST traceable where available.

5.2.1 Standard Logbooks. See the Reagent and Standards Tracking SOP for instructions on ensuring traceability.

5.2.2 Use of Volumetric Glassware. Standards must be prepared volumetrically using class A volumetric glassware or calibrated eppendorf pipettes. Do not use disposable pipettes to prepare standards.

5.2.3 Standard Storage. Store all standards at room temperature. Prepared standards or spiking solutions may be kept for up to one month. Purchased solutions may be kept until the manufacturers expiration date as long as storage requirements for the solution are followed. Replace standards sooner if it is suspected that the standard has degraded or concentrated.

5.2.4 Purchasing Standards. The recommended sources of the calibration and verification standards are listed in Table 2.

Table 2. Recommended Standard Sources

Analyte	Calibration Standards		Second Source Verification Standards	
	Vender / Catalog #	Conc (ug/mL)	Vender / Catalog #	Conc (ug/mL)
Mercury	CPI / 4400-1000331	1000	PE / N9300174	1000

5.2.5 Calibration Standards. The recommended concentrations for the calibration standards are given in Table 3.

Table 3. Recommended Calibration Standards and CCVS/LCS

Analyte	Blank	Level 1 (ppm)	Level 2 (ppm)	Level 3 (ppm)	Level 4 (ppm)	Level 5 (ppm)	CCVS/LCS (ppm)
Mercury	0.0000	0.0005	0.0010	0.0025	0.0050	0.0100	0.0025

5.2.6 Second Source Verification Standard (CCVS/LCS). Throughout this SOP, the second source verification standard is referred to as the CCVS/LCS. The CCVS/LCS must be prepared from different standards than those used to prepare the calibration standards. See Table 2 for recommended standard sources. The CCV/LCSS must be prepared at a concentration between 25% and 50% of the high

standard in the initial calibration curve. The recommended concentrations for each compound are given in Table 3.

5.2.7 MS/MSD spiking solution. It is recommended that MS/MSDs be prepared using the same standard that is used to prepare the CCVS/LCS. The recommended concentrations for each compound in the MS/MSD are the same as shown for the CCVS/LCS in Table 3.

6.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

It is recommended that non-aqueous samples be collected in clear glass containers. The minimum sample amount required for analysis is less than 1 gram. It is recommended that a 4oz jar of sample be collected at the time. Store all soil and waste samples refrigerated at 4°C from the time of collection until digestion and analysis.

The holding time for metals samples is 28 days from the date of sample collection.

7.0 QUALITY CONTROL

The following details the QC requirements that apply to this analysis. Each Quality Control Indicator (QCI) provides information pertaining to either method or individual sample performance. Our goal is to produce defensible data of known and documented quality.

7.1 Preparation Blank

7.1.1 Definition & Use of the Preparation Blank

For the purposes of this SOP the preparation blank is defined as a 8oz bottle which is taken through the entire digestion process. Untreated preparation blanks are not performed on a solid matrix because of the difficulty in finding a metals free solid matrix. The purpose of the preparation blank is to demonstrate that the entire analytical process, including the digestion and analytical procedure, is free from sources of contamination. For this reason, the blank must be taken through the entire analytical process used for the associated samples, including digestion.

7.1.2 Frequency of the Preparation Blank

A preparation blank must be digested with each preparation batch of 20 samples or less. Analyze a preparation blank with each analytical batch of 10 or less, and then before and after every tenth sample.

7.1.3 Criteria for the Preparation Blank

Evaluate the preparation blank for mercury. If mercury is present above the reporting limit the blank is out of control.

7.1.4 Corrective Action for the Preparation Blank

If a preparation blank does not meet the above criteria, then the concentration of the blank versus the samples in the associated preparation batch will need to be compared.

If the concentration of the sample is greater than 10 times the level in the blank, the sample can be reported with a "B" flag indicating blank contamination.

If the concentration of the sample is less than 10 times the level in the blank, the sample is out of control and should be re-digested. If this is not possible (i.e., lack of sample or holding time has expired) the sample must be reported with a "B" flag indicating blank contamination.

Clean samples without hits can be reported without qualifiers.

In all cases the amount detected in the blank should be listed on the data report and the source of contamination should be identified and eliminated to prevent the problem from continuing.

7.1.5 Documentation for the Preparation Blank

All hits above the reporting limit must be clearly indicated in the raw data. All preparation blanks should be filed in a manner that will ensure that they can be easily retrieved and referenced to all associated samples.

7.2 Laboratory Control Sample (CCVS/LCS)

7.2.1 Definition & Use of the CCVS/LCS

The CCVS/LCS is a spike performed in the BOD bottle. Non-aqueous LCSs are not performed in a solid matrix because of the difficulty in finding a reliable blank solid matrix. The concentration of the CCVS/LCS must be 20% to 80% of the maximum of the calibration range. CCVS/LCSs are carried through the entire analytical process that any of the associated samples go through, including digestion. The standard used to spike the CCVS/LCS must be from a different source or a different lot number than the calibration standards.

For the purposes of this SOP, the CCVS/LCS fulfills the requirements for the CCVS, the LCS, and the ICVS.

7.2.2 Frequency of the CCVS/LCS

A CCVS/LCS must be digested with each preparation batch of 10 samples or less.

Analyze a CCVS/LCS immediately following every calibration curve, and then before and after every tenth sample.

7.2.3 Criteria for the CCVS/LCS

CCVS/LCS results must meet the criteria given in Attachment A. The statistical criteria in Attachment A must meet or exceed 90% - 110% recovery.

7.2.4 Corrective Action for the CCVS/LCS

If a CCVS/LCS fails criteria as described above, suspend the analysis and immediately re-analyze the CCVS/LCS once. If the re-analysis is in control, then sample analysis may continue. If the re-analysis is out of control, the analysis must stop and corrective action must be taken as described below.

Determine the cause and correct the problem. Two consecutive passing CCVS/LCSs must be analyzed (i.e., no samples or QCIS in between) or else a new ICAL must be analyzed. All samples since the last in control CCVS/LCS must be re-digested and/or re-analyzed. Re-digestion is required if the problem was determined to be with the digestion. If this is not possible (e.g., not enough sample or holding time has expired), the sample data must be reported indicating that the LCS is out of control.

7.2.5 Documentation for the CCVS/LCS

The percent recovery or percent difference should be summarized in a table or report. The CCVS/LCS should be filed in a manner that will ensure it can be easily retrieved and referenced to all the associated samples. The ID number of the standard used to spike the CCVS/LCS should be recorded in the metals prep logbook and on the raw data.

7.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

7.3.1 Definition & Use of MS/MSD

The purpose of the MS/MSD is to confirm that the matrix being analyzed is not interfering with the recovery of mercury. MS/MSDs are defined as spiked client samples. Select samples to be spiked on a rotating basis from among various client samples, waste streams, and other applicable locations. MS/MSDs are carried through the same analytical processes that the unspiked native sample goes through, including digestion. MS/MSD data alone cannot be used to evaluate the precision and accuracy of individual samples except for the sample chosen for the MS/MSD analysis. MS/MSDs can also serve to monitor the long term precision and accuracy of the method.

7.3.2 Frequency of MS/MSD

A MS/MSD must be digested with every group of 20 samples or once per day, whichever comes first.

7.3.3 Criteria for MS/MSD

Calculate the RPD and percent recovery. The MS/MSDs must meet the criteria in Attachment B.

7.3.4 Corrective Action for MS/MSD

No action is taken on out of control MS/MSD data alone to qualify an entire batch. However, the data may be used in conjunction with other QC criteria to determine the need for qualifying the entire batch.

If the MS/MSD data is outside acceptance limits, check percent recovery for the CCVS/LCS. If the CCVS/LCS is in control, the procedure is in control and the preparation batch is in control. However, a potential problem exists with the sample that was spiked.

The corresponding unspiked native sample must be flagged with the "MS" flag to indicate that the MS/MSD was out of control but that the batch is in control.

If the MS/MSD set is outside RPD limits a potential problem exists with sample composition. The corresponding unspiked native sample must be flagged with the "P" flag to indicate that the MS/MSD failed RPD limits.

If the native sample is high in metals, the corresponding MS/MSD may require dilution to the point that the spike amount becomes insignificant. If the MS/MSD requires dilution to the point that the expected amount in the diluted aliquot would be less than the reporting limit, report the result as "diluted out" and flag with the "D" flag. For example, consider a MS/MSD that is spiked at 50 ug/L with a reporting limit of 2 ug/L. A 1/5 dilution of the MS/MSD would then result in an expected concentration in the diluted sample of 2 ug/L (plus the level in the native sample), which is the reporting limit reported. While a 1/50 dilution would be reported as "diluted out".

7.3.5 Documentation for the MS/MSD

The RPD and percent difference should be summarized in a table or report. The MS/MSD should be filed in a manner that will ensure it can be easily retrieved and referenced to the associated samples. The standard ID number of the standard used to spike the MS/MSD should be recorded in the metals prep logbook.

7.4 Reporting Limit Verification Standard (RLVS)

7.4.1 Definition & Use of RLVS

The Reporting Limit Verification Standard (RLVS) provides information regarding instrument performance at the reporting limit. The concentration of the RLVS must be at or below the reporting limit.

7.4.2 Frequency of RLVS

Analyze this standard after performing instrument calibration. In some cases, it may be possible to incorporate the RLVS into the calibration curve.

7.4.3 Criteria for RLVS

Acceptance criteria requires the percent recovery to be within [illegible] of the true value.

7.4.4 Corrective Action for RLVS

An out of control RLVS indicates an improperly prepared standard or potential problems with the calibration curve. If the RLVS is too high but samples are less than the client reporting limit, then data is considered acceptable and can be reported. Otherwise re-analyze the RLVS to confirm the [illegible] result. If the RLVS is still out of control, identify and correct the source of the problem. Re-calibrate and re-analyze the affected samples.

7.4.5 Documentation for RLVS

Record the percent recovery and the ID number of the RLVS in the raw data.

7.5 Reagent Blank.

Not Applicable - Preparation blanks are used in place of reagent blanks when performing non-aqueous mercury analysis. For the purposes of this SOP all the QC is brought through the digestion procedure.

7.6 Linear Dynamic Range (LDR) Study.

7.6.1 Definition and Use of the LDR

The LDR is defined as the concentration range over which the analyte calibration remains linear. The LDR is determined by analyzing successively higher standard concentrations (at least five) until the [illegible] signal exceeds the criteria given below.

7.6.2 Frequency of LDR

A LDR study must be performed during instrument validation prior to running client samples. For those analytes that periodically approach the upper limit of the range, the range should be verified every six months. The LDR shall also be verified whenever a significant change is made to the method, the instrument, or the operating conditions.

7.6.3 Criteria for LDR

Determine the upper limit of the linear dynamic range by analyzing successively higher standards until the observed percent recovery is not within 90% - 110% of the true value. The standard level just below the one that fails is the upper limit of the linear dynamic range.

The LDR can be subsequently verified by analyzing a standard at the determined LDR concentration. If the observed percent recovery of the standard is within 90% - 110% of the true value, then the LDR is still valid.

7.6.4 Corrective Action for LDR

By definition the LDR is the standard level just below the one that fails and therefore there is no applicable corrective action after attempting to determine the LDR.

If recovery criteria is not met during an attempt to verify a previously determined LDR, the LDR is invalidated and a new LDR study must be performed to determine the new upper limit of the linear dynamic range.

7.6.5 Documentation of LDR

The LDR (and any subsequent verifications done on the LDR standard) must be kept in a manner that will ensure that it can be easily retrieved by the analyst. The ID number of the LDR standard must be recorded in the raw data.

7.7 Initial Calibration Curve (ICAL)

7.7.1 Definition & Use of the Initial Calibration Curve

The purpose of the ICAL is to relate detector response to sample concentration. It also provides a way of verifying that detector response, over a predetermined concentration range, can be predicted using a mathematical equation. If the response were erratic, there would be no accurate way to relate response to concentration and the curve would not pass. The minimum of a blank and five standards are required to construct the calibration. The initial calibration curve will be used to quantitate samples and QCIs.

The initial calibration curve for non-aqueous mercury analysis is brought through the entire process used for samples, including the digestion procedure.

7.7.2 Frequency of the Initial Calibration Curve

An initial calibration curve must be analyzed daily. It may also need to be re-analyzed during the corrective action process when analytical QC fails to meet acceptance criteria (such as the CCVS/LCS or reagent blank).

7.7.3 Criteria for the Initial Calibration Curve

The correlation coefficient must be 0.995 or greater for the initial calibration curve to be acceptable.

7.7.4 Corrective Action for the ICAL

Since the initial calibration curve is used to calculate results, an analyte cannot be reported until the above criteria is met. Perform the necessary corrective actions and re-analyze the initial calibration curve along with any samples or QC that were analyzed using the out of control calibration.

7.7.5 Documentation for the ICAL

The correlation coefficient should be summarized in a table or report. The ICAL should be filed in a manner that will ensure it can be easily retrieved and referenced to all associated samples. Record the standard ID number of the ICAL standards on the raw data

7.8 Initial Calibration Verification Standard (ICVS)

See CCVS/LCS.

7.9 Continuing Calibration Verification Standard (CCVS)

See CCVS/LCS.

7.10 Method Detection Limit (MDL) Study.

A MDL study must be done during initial method validation and then it must be verified annually. If the analytical method is changed, a new MDL study must be performed. MDLs must be brought through the entire analytical process including digestion. Follow the Procedure for Detection Limit Studies SOP when performing and evaluating the MDL study.

7.11 Method Validation Study (MVS).

7.11.1 Definition & Use of MVS

The purpose of the MVS is to verify that the method can generate precise and accurate analytical data. The MVS consists of four replicate spikes that are brought through the entire analytical process including digestion. Non-aqueous LCSs are not performed in a solid matrix because of the difficulty in finding a metals free solid matrix. MVSs should be spiked at approximately 10 times the calculated MDL. The standards used to prepare the MVS must be from a different source or a different lot number than the standards used to prepare the initial calibration curve. MVSs are used to validate new analysts and new instruments, and to validate changes in the analytical equipment or techniques.

7.11.2 Frequency of MVS

A set of method validation samples must be analyzed, at least once, by each analyst performing this method. A set of method validation samples must be analyzed on each instrument that will be used to perform this method. However, each analyst does not have to analyze a set of method validation samples on every instrument. Method validation must be performed whenever a significant change in the method or instrumentation is made which could cause the previous MVS to become invalidated.

7.11.3 Criteria for MVS

The average percent recovery must be within 80% - 120% of the true value and the %RSD must be less then or equal to 20%.

7.11.4 Corrective Action for MVS

If a MVS fails to pass the criteria in section 7.11.3, the problem must be identified and corrected. If the problem is related to the instrument, re-analyze the MVS after correcting the problem. If the problem is related to digestion, re-digest and then re-analyze the MVS after fixing the problem. An acceptable MVS must be performed prior to analyzing samples.

7.11.5 Documentation for MVS

One method validation study may serve several purposes including analyst training, method validation, and instrument validation. The MVSs should be filed in a manner that will insure that they can be easily retrieved for all intended purposes. The ID number of the standard used to spike the MVS must be documented in the metals prep logbook.

7.12 Analyst Certification

This method is restricted to use by, or under the supervision of, analysts trained in the use of the FIMS as a qualitative and quantitative tool. Prior

to performing this analysis, each analyst must successfully complete the requirements detailed in the Analyst Certification SOP.

8.0 PROCEDURE

8.1 Sample preparation

8.1.1 Transfer three 0.2 g portions of sample (three aliquots of sample from different areas of the jar) to a single 300mL BOD bottle. The intent here is to ensure the sample aliquots are as representative of the entire sample as possible.

8.1.2 Add 5mL of reagent water and 5mL of aqua regia.

8.1.3 Add 50mL of reagent water and 15 mL of 5% potassium permanganate solution to each sample bottle. Heat in the water bath at 95°C for 2 hours.

8.1.4 **Caution: Perform this step under a hood as Cl_2 may be evolved.** Allow to cool and add 50 mL of reagent water. Then add 6mL of sodium chloride hydroxylamine hydrochloride to reduce the excess permanganate.

8.1.5 Add an additional 50uL of the 0.5% potassium permanganate. The sample is now ready to be transferred to the autosampler for analysis.

8.2 Analytical Preparations.

8.2.1 Prior to beginning analysis, set up the flow injection system according to the manufacturer's instructions. The analytical wavelength used must be 253.7 nm. Allow the instrument to warm up for at least one hour prior to analysis.

8.2.2 Refer to Table 4 for the instrument operating conditions. Changes to this programming must be approved by the laboratory management and Quality Assurance Coordinator. Certain changes in programming will require repeat demonstration of capability studies.

Table 4. Instrument Operating Conditions

Integration Time	20
Data Processing	Peak Height, Smoothing: 0.5 sec or 19 points
Lamp	HCL or EDL
Slit (nm)	0.7 (Low or Alt)
Wavelength (nm)	253.7
Cell Temperature	100°C

Carrier Solution	3% (v/v) HCl
Reducing Agent	1.1% $SnCl_2$ in 3% (v/v) HCl
Carrier flow	70 - 100 mL/min

8.3 Replicate Measurements

For the purposes of this test, all analyses are performed in duplicate and the results are averaged. For all standards (calibration standards, MDLs, LDRs, etc.) the two replicate runs must agree within 5% RSD. No %RSD criteria is used for blank results because of the high variability when very little analyte is present. For all other runs including samples, sample QC (MS/MSL, MVS, MDL, etc.) and the RLVS the two replicate runs must agree within 20% RSD.

If the above criteria is not met, re-analysis is required. Some sample matrices may require dilution in order to bring the replicate %RSD into control. When dilution is required the reporting limits will have to be raised accordingly. Flag the sample results with the "MX" flag to indicate that a dilution was required due to the sample matrix.

In all cases the average result from the replicate measurements is used for reporting and evaluating QC.

8.4 Instrument Maintenance

8.4.1 At the end of each day all tubing should be flushed with reagent water and then air.

8.4.2 Release the pump tension on all tubing when turning off the instrument.

8.5 Initial Calibration Curve

The initial calibration curve consists of a minimum of a blank and five standards analyzed at different concentrations. Section 9.2 further defines the definition, frequency, criteria, corrective action, and documentation required for the initial calibration curve.

8.5.1 Calibration Technique. The only calibration technique which can be used for this method is a linear calibration which is not forced through zero.

8.5.2 Calibration and CCVS/LCS. Spike a series of BOD bottles with the appropriate amount mercury for each calibration standard and the CCVS/LCS. The volume of spike used will create a slight discrepancy in the final volume of the standard and therefore the maximum spiking volume allowed is 1mL. A spiking volume of 1mL will result in less than a 2% error in the standard. If a higher concentration standard is needed, start with a more concentrated spiking solution. After spiking the BOD bottles, process the standards as in sections 8.1.2 through 8.1.5 (including digestion).

8.5.3 Quantitation With The ICAL. In all cases the calibration curve is used to quantify samples and QCIs. An initial calibration curve must be analyzed and evaluated before any hit for that analyte can be quantitated.

8.5.4 Analysis of ICAL. The ICAL must be analyzed under the same conditions that will be used to analyze samples.

8.5.5 Calibration Verification. The CCVS/LCS must be analyzed to verify that the calibration remains valid over the course of the day. Analyze the CCVS/LCS using the same procedures used for the ICAL. CCVS/LCSs are subjected to the same procedure used for samples, including digestion. See section [illegible] for the frequency, criteria and corrective action for CCVS/LCSs.

8.6 Rinse Time (Rinse Blank).

Rinse the system with reagent water prior to the analysis of each sample. The rinse time will be one minute. This is sometimes referred to as a rinse blank. Shorter rinse times may be validated using the following procedure.

Aspire a standard containing each element at a [illegible] of the established LDR. The aspiration time for this standard should be the same as the normal sample analysis period. After aspirating the standard, analyze a series of reagent blanks at desired intervals. The length of time required to reduce the analyte signals to within a factor of 2 times the established MDL is noted. This time is then used as the minimum required rinse period.

Document the rinse time study and file in a location that can be easily retrieved for future reference.

8.7 Sample Analysis

8.7.1 Instrument Reagents. Prepare the standards and rinse solution as indicated in section 5.1. Feed this reagent into the [illegible] bottle on the instrument.

8.7.2 Sample Analysis. Each analytical batch can contain up to 20 samples. See section 1.1 for a more detailed definition of analytical batch.

8.7.3 Preparation Blanks. Preparation blanks must be subjected to the same digestion procedures used on the samples (sections 8.11 through 8.11.4). A preparation blank must be digested with each preparation batch. Analyze a preparation blank with each calibration curve, and then before and after every tenth sample.

8.7.4 Matrix Spike and Matrix Spike Duplicate (MS/MSD). A MS/MSD must be included with every group of 20 samples or once per day, whichever comes

first. MS/MSDs must be subjected to the same digestion procedures used on the samples (sections 8.1.2 through 8.1.5).

9.0 CALCULATIONS/DATA REDUCTION AND INTERPRETATION

9.1 Qualitative analysis

The first step in evaluating the data is qualitative analysis. Qualitative interpretation involves identifying the specific analytes that are present. Analyte identification is based upon observed sample absorbance at the specified wavelength.

9.2 Quantitative analysis

The second step in evaluating data is quantitative analysis. A quantitation is performed using the initial calibration curve.

9.2.1 Calculations. Use the average result from the replicate injections to perform all calculations. Calculate the concentration in the sample as follows:

$$\text{Concentration (ug/g or mg/Kg)} = \frac{(C) * (V_f)}{W_i}$$

where:

C = The concentration result reported by the instrument in µg/L.
V_f = Final volume of digestate (in liters).
W_i = Total weight of sample used (in grams).

9.2.2 Calculating the True Value of Spikes. The following equations must be used to calculate the true concentration of an analyte in a MVS or matrix spike.

SPIKE CONCENTRATION (TRUE VALUE) IN SOIL

$$\text{Spike concentration (true value) in Soil (ug/g)} = \frac{M}{W}$$

Where:

M_s = The mass of analyte (in ug) spiked onto the sample or QC.
W_s = Initial weight of sample or QC (volume digested in grams).
For QCs assume a nominal weight of 0.5 grams.

9.2.3 Calculating Relative Percent Difference (RPD). Use the following equation to calculate RPD on MS/MSDs:

$$RPD = \left| \frac{(A - B)}{C} \right| \times 100$$

Where: A = MS result (in concentration NOT percent recovery)
B = MSD result (in concentration NOT percent recovery)
C = Average of the MS and MSD results

9.2.4 Calculating Percent Relative Standard Deviation (%RSD). Use the average and the standard deviation to calculate percent relative standard deviation (%RSD) according to the following equation:

$$\%RSD = \frac{\text{standard deviation}}{\text{average}} \times 100$$

9.2.5 Dilutions. Dilution is required if any sample exceeds the upper limit of calibration. Compare diluted results with the results of the original analysis to verify that the results seem reasonable before reporting.

9.3 Reporting of Results.

9.3.1 Significant Figures. Report all results to two significant figures except for MS/MSDs which are reported to three significant figures.

10.0 METHOD PERFORMANCE

Laboratory performance criteria for both precision and accuracy are provided in section 7 of this SOP. These limits are defined by the method and required to demonstrate initial method performance. The most recent method validation and detection limit studies are kept on file in the laboratory and can be provided upon request.

Further, ongoing CCVS/LCS and MS/MSD limits are provided in the attachments to this SOP. These limits were developed through the statistical evaluation of QC recoveries.

11.0 POLLUTION PREVENTION

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based upon anticipated usage and reagent stability).

12.0 WASTE MANAGEMENT

Laboratory waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description codes and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the Waste Disposal SOP.

13.0 REFERENCES

13.1 Perkin Elmer Method 245.1: Determination of Mercury in Drinking Water and Wastewater by Flow Injection Atomic Absorption Spectrometry (Cold Vapor Technique), Perkin Elmer 1992.

13.2 Test Methods for Evaluating Solid Waste, 3rd Edition.

14.0 METHOD DEVIATIONS

Deviations from the referenced Methods and IL Part 186 (NELAC) requirements are as follows:

14.1 NELAC does not allow extrapolation of results either below or above the calibration curve. This SOP allows extrapolation of the results below the lowest point of the curve. However, this SOP requires the analysis of a Reporting Limit Verification Standard (RLVS) at or below the reporting limit to verify the accuracy of low level results.

14.2 This SOP does not require IDL determinations. All determinations relating to sensitivity are performed using the MDL or equivalent.

14.3 Section 7.1 of Method 7471A requires that 55 mL of reagent water be added to the digestate prior to analysis. This appears to be [illegible] compensate for a different final volume in the samples versus the standards. The method only states to add 50 mL of reagent water to the standard digestates but has you add an extra 5 mL of reagent water to the standards prior to digestion. The procedure in this SOP only calls for 50 mL of reagent water to be added to the digestates from both standards and samples. This creates a slight discrepancy between the final volume of standards versus samples but the total error is less than 2%.

14.4 Section 5.4 of Method 7471A states that a 10% solution of stannous chloride can be substituted for the stannous sulfate solution. It also states are given on how to prepare the 10% stannous chloride solution. This SOP incorporates the use of a 1.1% w/v stannous chloride solution that is prepared in a 3% hydrochloric acid matrix.

14.5 Section 7.1 of Method 7471A requires that the samples be heated at 95° for 2 minutes after the addition of aqua regia. This heating step is not included in this SOP.

14.6 Section 7.1 of Method 7471A states that after the 30 minute digestion, 6mL of sodium chloride hydroxlyamine sulfate is added to reduce the excess permanganate. Then 55mL of reagent water is added followed by the addition of stannous sulfate (or stannous chloride). This SOP changes the [illegible] these steps around as follows. First 50mL of reagent water is added instead of 55mL). Then 6mL of sodium chloride hydroxlyamine sulfate is added. This SOP incorporates an extra step where an additional [illegible] of potassium permanganate is added after the addition of the sodium chloride hydroxlyamine sulfate.

14.7 Section 8 of Method 7471A refers the user to Section 8 of Method 7000A for quality control. Section 8.4 of Method 7000A requires that MS/MSDs be performed "with each analytical batch". However, the method does not define

exactly what is meant by an analytical batch. This SOP requires a MS/MSD daily or with every 20 samples, whichever comes first.

14.8 Section 8.6 of Method 7000A requires a serial dilution test be performed for at least one sample in each analytical batch. This SOP does not include a serial dilution test.

14.9 Section 8.6.2 of Method 7000A requires that post digestion spikes (recovery test) be performed when MS or MSDs fail recovery criteria. If the post digestion spike does not meet recovery criteria, section 8.7 of Method 7000A requires the use of MSA to calculate results. This SOP does not require post digestion spikes or the use of MSA. If MS or MSD results are out of control, the data for the sample that was spiked is flagged.

Attachment A
Metals - Mercury LCS Criteria

	Drinking Water [1]	Aqueous (GW & WW) [1]	Non-aqueous [1]
Mercury	91.2% - 108.5%	80.0% - 120.0% [3]	80.0% - 120.0% [2]

1. Statistical limits developed 2/1/00.

2. Not enough data was available to develop limits, therefore the method limits are used.

3. Statistical limits did not meet or exceed method requirements, therefore the tighter limits from the method are used.

QA Approval/Date:

Bonnie Hadelman / 2/11/00

Analyst Signature/Date:

[signature] / 2/14/00	_____ / _____
Virginia S Medeck / 2/16/00	_____ / _____
Emily F Wadley / 8/10/00	_____ / _____
_____ / _____	_____ / _____
_____ / _____	_____ / _____
_____ / _____	_____ / _____

Attachment B
Metals - Mercury MS/MSD Criteria

	Drinking Water [1]		Aqueous (GW & WW) [1]		Non-aqueous [1]	
	Percent Recovery	RPD	Percent Recovery	RPD	Percent Recovery	RPD
Mercury	78.7% - 120.6%	<20.0 [3]	75.0% - 125.0% [3]	<20.0 [3]	75.0% - 125.0% [2]	<20.0 [2]

1. Statistical limits developed 2/1/00.

2. Not enough data was available to develop limits, therefore the method limits are used.

3. Statistical limits did not meet or exceed method requirements, therefore the tighter limits from the method are used.

QA Approval/Date:

Bonnie Hadden and 2/11/00

Analyst Signature/Date:

[signature] / 2/14/00	_____ / _____
[signature] / 2/16/00	_____ / _____
[signature] / 2/10/00	_____ / _____
_____ / _____	_____ / _____
_____ / _____	_____ / _____
_____ / _____	_____ / _____

TestAmerica Inc.

Bartlett Division
850 West Bartlett Rd
Bartlett, IL 60103

Standard Operating Procedure

Analyte or Suite: pH - Soil and Waste (<20% water by volume)

Methodology: Potentiometric Electrode w/ Temperature Correction

Reference: SW-846, 3rd Edition, Method 9045C

Revision # 5 Date revised: July 4, 1999

File name: /usr3/sops/sop.9045c.5

Approvals:

Division Manager

Operations Manager

Quality Assurance Coordinator

Technical Manager

SOP REVIEW

I have read and reviewed this SOP for accuracy with actual laboratory procedures. I understand and agree to follow the procedures in this SOP. I acknowledge that any deviations from the SOP must be approved by management in writing unless otherwise indicated in the SOP.

Print Name	Signature	Date
Laura Moen-Hacek	Laura Moen-Hacek	7/6/99
Katie Thurkill	Katie Thurkill	7-11-00

Table of Contents

1. Introduction and Scope

1.1. General

1.1. This method is used to measure the pH of soil, and waste samples. Wastes may be solids, sludges or nonaqueous liquids. If water is present it must constitute less than 20% of the total volume of sample.

1.2. Definitions

EPA: means the United States Environmental Protection Agency.

NIST: The United States Department of Commerce, Technology Administration, National Institute of Standards and Technology same instrument.

Quality Control Indicators (QCIs) - A general term used to refer to blanks, duplicates, etc...

Second Source: means a different vendor or manufacturer, or different lots of the same vendor or manufacturer.

Wastes: Wastes are considered solids, sludges, or non-aqueous wastes.

2. Summary of Method

The pH of the sample is determined electrometrically using a combination electrode with automatic temperature compensation. The measuring device is calibrated using a series of standard solutions of known pH. The sample is mixed with reagent water, and the pH of the resulting aqueous mixture is measured.

3. Safety

Employees should comply with all safety policies as presented in the Safety Manual. Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards, and all associated protective wear and spill clean up procedures prior to the use of any chemical. This information can be obtained by reviewing the applicable material safety data sheet (MSDS). The bottle labels also provide important information that must be noted. If you have any questions consult your supervisor or safety officer.

Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods. It should be noted that samples must be handled with as much (or more) care as any of the materials used in this method due to the unknown nature of their composition.

4. Apparatus and Materials

4.1. Apparatus

The following apparatus is recommended for performing this procedure. Equivalent items can be used, if with their use, the analytical and QA/QC requirements in this SOP can be met.

4.1.1. Orion Model 410A or Accumet 25

4.1.2. Magnetic stirrer and Teflon-coated stirring bar.

4.1.3. Thermometer or temperature sensor for automatic compensation (recommended model has this feature.)

4.1.4. Disposable beaker cups

4.1.5 Top-Loading Balance, capable of weighing to 0.1 g.

4.2 Reagents

The following reagents are required to perform this procedure. When instructions are given on how to prepare a specific volume of a reagent, larger or smaller volumes can be prepared as needed so long as the final concentrations remain the same. Any other deviation from the reagents listed in this SOP could be detrimental to the quality of the data produced. Such deviations would have to be approved and documented in the SOP. All reagents must be properly labeled with the reagent identification and date received, expiration date, initials of the analyst, and applicable safety information.

4.2.1. Reagent Traceability. With the exception of deionized water, traceability of the following reagents must be documented as described in the "Standards/Reagents Documentation And Tracking" SOP.

4.2.2. Deionized water. Prepare by passing water through a mixed bed of cation and anion exchange resins or an equivalent source. Use deionized water for the preparation of all reagents, calibration standards, and dilution water. Deionized water is needed for rinsing the electrode.

4.2.3. Hydrochloric acid (1:10). Pour approximately 800 mL of deionized water into a 1 L volumetric flask. Add 100 mL of concentrated hydrochloric acid. Dilute to mark with deionized water. This reagent is only needed for cleaning the electrode and the acid strength is not particularly critical so long as it is close to 1:10. For example, 1N HCl could be used instead of 1:10 HCl. Shelf life = 1 year.

4.3 Standards Preparation

The following standards are recommended for performing this procedure. The use of alternative standards will be allowed as long as the analytical and quality control objectives of the method can be met. When instructions are given on how to prepare a specific volume of standard, larger or smaller volumes can be prepared as needed. Standards must be NIST or EPA traceable when available.

4.3.1. Standard Traceability. See the "Standards/Reagents Documentation And Tracking" SOP for instructions on ensuring traceability.

4.3.2. Standard Storage. The original (purchased) buffer solution is good until the expiration date provided by the manufacturer or a year from the date of purchase, whichever comes first. If an aliquot of the buffer solution is put into another bottle, the aliquot is good for one month. Aliquots of buffer solution that were analyzed (i.e., such as to calibrate or to check calibration) should be discarded immediately after use. NEVER put anything, including the pH electrode, into the original standards bottles.

4.3.3. Calibration Standards. Buffer solutions having pH of 4.00, 7.00, and 10.00 are used for routine calibration and calibration verification. Additional buffer solutions having a pH of 2.00 and 12.00 are necessary when samples are not bracketed by the normal calibration range. Colored solutions are available in bulk and are convenient to our application.

4.3.4. Initial Calibration Verification Standard (ICVS). The ICVS is a second source standard used to ensure the validity of the calibration buffers/standards. A second source buffer solution at pH 7.40 is available for confirming calibration.

5. Interferences

5.1. The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or high salinity.

5.2. At the low and high ends of the pH scale, the standard glass pH electrode does not perform accurately. Specialty electrodes are available from some manufacturers. At pH greater than 10, sodium affects pH, yielding an erroneously low result. At pH less than 1, a standard glass electrode will yield erroneously high values. An appropriate electrode for use in these pH ranges will specify low sodium error for high alkalinity and a membrane feature for low pH readings.

5.3. Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by rinsing with distilled water. An additional treatment with 0.1N hydrochloric acid or methanol may be necessary to remove any remaining film.

5.4. Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This error is sample-dependent and cannot be controlled. It should, therefore, be noted by recording both the pH and temperature at the time of analysis.

5.5. The general application of the pH paper test is to dip a portion of the sample onto a pH test strip and comparing the color indicator bands to the reference chart translating color responses to pH values. The pH paper method is more susceptible to interferences than the electrode method.

5.6. The pH paper method is not a substitute for the electrode method except in those cases in which the sample will adversely affect or damage the electrode. Use the pH paper method only for oily organic materials, inks, paints and spot tests for complete unknowns prior to applying the electrode.

6. Analytical Procedure

6.1. Sample Collection, Preservation, and Handling

6.1.1. Samples must be measured as soon as possible after collection. Maximum sample holding time is 7 days from sample receipt.

6.1.2. No preservative is required. Samples should be kept cool (4°C) during transportation to the lab.

6.2. pH Meter Operation and Maintenance.

6.2.1. Operation. Because of the wide variety of pH meters and accessories, detailed operating procedures are not incorporated into this SOP, see Attachments. Each analyst must be acquainted with the operation of each system and familiar with all instrument functions.

6.2.2. Electrode Maintenance. Special attention to care of the electrodes is necessary to keep the pH meter in good working order.

6.2.2.1. The internal solution level must be maintained above the internal element at all times. Fill whenever low.

6.2.2.2. The sensing bulb and junction must not be allowed to dry out. Electrode must be stored at all times when not in use in pH 7.0 buffer. Refer to the product manual for the recovery procedure should you need to recover a dried-out electrode.

6.2.2.3. Any pH maintenance, change in electrode or abnormalities should be written in pH maintenance log book.

6.2.2.4. Coatings of oily material or particulate matter can be removed by washing with detergent, rinsing several times with water, soaking the lower 1/3 of the electrode in 1:10 HCl, and then thoroughly rinsing with water.

6.2.3. Stir Plate. A magnetic stir plate is used to mix buffer solutions during analysis (soil and waste samples are not mixed during analysis). The stir plate becomes warm after long periods of use and therefore the buffer solutions must be insolated from this warming effect during analysis. This can be easily accomplished by suspending the disposable sample cup in a beaker on the stir plate. The magnetic stir bar in the bottom of the cup will still turn, but the bottom of the cup will not be in contact with the warm surface of the stir plate. Failure to do this will likely result in erratic results and difficulty in passing calibration criteria.

6.3. Instrument Calibration.

6.3.2. Initial Calibration (ICAL). DAILY, each instrument/electrode system must be calibrated with a minimum of three points that bracket the expected pH of the samples and are approximately two pH units or more apart. Buffers of ph 4.00, 7.00, and 10.00 are normally used. Follow the manufacturer's instructions to perform the 3 point calibration. After completing the initial calibration read each of the 3 buffers back against the calibration to evaluate acceptability. See section 7.3 for criteria and corrective actions.

6.3.3. Initial Calibration Verification Standard (ICVS). Each calibration must be verified with a buffer solution from an independent source (external standard or ICVS). See section 7.4 for criteria and corrective actions required for the ICVS.

6.3.4. Continuing Calibration Verification Standard (CCVS). The pH meter must be checked with a CCVS after every tenth sample. Normally the analyst should alternate between using the pH 4.00 buffer and the pH 7.00 buffer for CCVS analysis. If any samples have a measured pH of less than 4.0, the 2.00 buffer must be incorporated as a CCVS. If any samples have a measured pH of greater than 10.0, the 12.00 buffer must be incorporated as a CCVS. See section 7.5 for criteria and corrective actions relating to CCVSs.

6.4. Sample Analysis.

6.4.1. Follow the instrument manufacturer instructions for performing sample analysis after calibrating the instrument.

6.4.2. The following is the order of standard and sample analysis:

1. Calibrate using a minimum of 3 standard buffers
2. Analyze the 3 ICAL buffers to evaluate ICAL acceptance
3. ICVS
4. Analyze 10 samples
5. 10th sample duplicate analysis
6. CCVS
7. Analyze samples 11-20
8. 20th sample duplicate analysis
9. CCVS
10. etc.....always end analysis with a CCVS

6.4.3. Weigh 20 g of soil or waste sample. Add 20 mL of deionized water. Cover and continuously stir or shake for 5 minutes.

6.4.4. Problematic Matrices.

6.4.4.1. An additional dilution is allowed (20g to 40mL) if working with hydroscopic soils and salts or other problematic matrices. Documentation that the sample required an additional dilution and why it was needed is required in the lab book.

6.4.4.2. If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may then need cleaned as in section 6.2.2.4.

6.4.5. Soil samples must be allowed to sit for at least 1 hour so that all of the fine particles can settle. Wastes, sludges, and non-aqueous liquids must be allowed to sit for at least 15 minutes. Alternatively the analyst can filter or centrifuge off the aqueous phase of the sample for pH measurement.

6.4.6. Immerse the electrodes into the supernatant just deep enough to establish a good electrical contact through the ground glass joint or the fiber capillary hole.

6.4.7. Temperature correction is required for pH measurement if the sample temperature differs by more than 2°C from the buffer solution temperature during calibration. The pH meter is able to adjust for temperature automatically so long as this option is selected and a temperature electrode is installed.

6.4.8. Samples with a pH of 11 or greater must be analyzed at 25 +/- 1°C. A Water Bath can be used to hold samples at this temperature.

6.4.9. Samples with pH values of less than or equal to 2.00 and greater than or equal to 12.00 must be analyzed in duplicate.

6.4.10. The electrodes must be thoroughly rinsed in between samples.

7. Quality Control.

The following details the QC requirements which apply to this analysis. Each Quality Control Indicator (QCI) provides information pertaining to either instrument performance, method performance (including sample preparation), or individual sample performance. Our goal is to produce data of unquestionable quality.

7.1 Method Detection Limit

MDLs are not applicable to this method.

7.2. Analyst Certification.

Each analyst performing this method must successfully complete the requirements detailed in the certification SOP. Analysts demonstrate proficiency by analyzing a single or double blind performance sample.

7.3. Initial Calibration (ICAL).

7.3.1. Definition and Use of the ICAL

The purpose of the initial calibration is to ensure that the meter can accurately read pH values over the range of the analysis. Three calibration buffers are utilized to prepare that ICAL, a pH 4 buffer, a pH 7 buffer, and a pH 10 buffer. This brackets the normal range of pH values observed in the samples. Should any samples be encounted that do not fall within this range, buffers at pH 2 and at pH 12 are also available to analyze as CCVSs. Section 6.2 describes the procedure for initial calibration of the pH meter.

7.3.2. Frequency of ICAL

The ICAL must be performed daily prior to beginning analysis.

7.3.3. Criteria for ICAL

After performing initial calibration, read each of the three buffers back against the ICAL. The results must be within 0.05 pH units of the actual buffer value in order to be acceptable. Be sure to adjust the true value of the buffer for temperature. Typically a chart is provided on the bottle that will give the true values at various temperatures.

7.3.4. Corrective Action for ICAL

The ICAL must pass criteria before analysis can proceed. If any of the 3 calibration buffers do not read back within 0.05 pH units of their true value recalibration is necessary. Special attention to care of the electrodes is necessary to keep the pH meter in good working order. Refer to manufacturer's instructions for maintenance of the meter and the electrodes.

7.3.5. Documentation of ICAL

In the logbook, record the standards tracking numbers of each ICAL buffer, the true values of each ICAL buffer (making any necessary adjustments for temperature), and the pH values as read off the meter after calibration. Also document any corrective actions that were necessary.

7.4. Initial Calibration Verification Standard (ICVS).

7.4.1. Definition and Use of ICVS

The purpose of the ICVS is to verify that the buffers used for initial calibration of the meter were chemically pure, prepared properly, and that they have not degraded significantly since the time they were made. The ICVS must be obtained from a different source or be of a different lot number than the buffers used for initial calibration.

7.4.2. Frequency of ICVS

Analyze the ICVS immediately following initial calibration.

7.4.3. Criteria for ICVS

Results must agree within 0.1 pH units of the true value. Be certain to adjust the true value of the ICVS for variation due to temperature. Typically a chart is provided on the bottle that will give the true values at various temperatures.

7.4.4. Corrective Action for ICVS

Acceptable results for the ICVS must be obtained prior to analyzing and reporting samples. If the criteria cannot be met, re-evaluate the meter calibration. Verify the acceptability of the source used for the ICVS. Examine the meter and electrode to see if maintenance or cleaning is necessary.

If the problem is determined to be with the meter or with the ICAL, perform any necessary maintenance and then re-calibrate the meter prior to re-analyzing the ICVS. If the problem is determined to be with the ICVS (e.g., buffer solution was expired) then recalibration may not be necessary. Correct the problem with the ICVS and re-analyze to verify compliance.

7.4.5. Documentation

In the logbook, record the standards tracking number of the ICVS buffer, the true value of the ICVS buffer (making any necessary adjustments for temperature), and the pH value as read off the meter. Also document any corrective actions that were necessary.

7.5. Continuing Calibration Verification Standard (CCVS).

7.5.1. Definition and Use of CCVS

Normally the analyst should rotate between using the 4.00 buffer and the 7.00 buffer from calibration to analyze the CCVS. If there are samples that have a pH lower than 4.00, analyze the 2.00 buffer to verify that the calibration is accurate at lower pH values. If there are samples that have a pH greater than 10.00, analyze the 12.00 buffer to verify that the calibration is accurate at higher pH values.

7.5.2. Frequency of CCVS

Analyze a minimum of one CCVS after every tenth sample and at the end of the batch.

7.5.3. Criteria for CCVS

Results must agree within 0.1 pH units of the true value. Be certain to adjust the true value of the CCVS for variation due to temperature. Typically a chart is provided on the bottle that will give the true values at various temperatures.

7.5.4. Corrective Action for CCVS

Acceptable results for the CCVS must be obtained or else all samples analyzed following the last in control CCVS will need to be re-analyzed. If the criteria cannot be met, re-evaluate the meter calibration. Verify the acceptability of the source used for the CCVS. Examine the meter and electrode to see if maintenance or cleaning is necessary.

If the problem is determined to be a bad or expired CCVS buffer, replace the buffer with fresh solution and re-analyze the CCVS to verify compliance. If the fresh buffer solution passes criteria no futher corrective action is necessary.

If the problem is determined to be with the meter or with the ICAL, perform any necessary maintenance and then re-calibrate the meter prior to re-analyzing the CCVS. All samples analyzed following the last in-control CCVS must then be re-analyzed.

7.5.5. Documentation

In the logbook, record the standards tracking number of the CCVS buffer, the true value of the CCVS buffer (making any necessary adjustments for temperature), and the pH value as read off the meter. Also document any corrective actions that were necessary.

7.6. Duplicate.

7.6.1. Definition and Use of Duplicate

The duplicate is a separate aliquot of sample which is subjected to the same conditions that a sample undergoes. This data alone cannot be used to evaluate the precision of individual samples except for the sample chosen for the duplicate analysis.

7.6.2. Frequency of Duplicate

Analyze a minimum of one duplicate per every ten samples.

7.6.3. Criteria for Duplicate

Acceptance criteria requires the duplicate relative percent difference (RPD) to be less than 20%.

7.6.4. Corrective Action for Duplicate

No action is taken on out of control duplicate data alone to qualify an entire batch. Action taken must be weighed carefully since it may be difficult to determine if poor precision is a result of sample non-homogeneity/uniqueness, method defects, or laboratory technique. However, the data may be used in conjunction with other QC criteria to determine the need for qualifying the data. If the duplicate data is outside acceptance limits, check CCVS results. If the CCVS is in control, the procedure is in control and the data is acceptable. Potentially, a matrix problem exists. Additional steps may be taken to determine the extent of the matrix interference. Refer to the "Data Assessment and Review" SOP for additional details.

7.6.5. Documentation

The data generated can be presented, if necessary, as a statement of precision for a particular analysis on a given matrix. Record the RPD of the duplicate in the logbook. Enter the pH measurement of the duplicate sample into the sample duplicate field in LABSYS.

8. POLLUTION PREVENTION

8.1 Pollution Prevention encompasses any activity or technique that reduces or eliminates the amount waste at the point of generation. There are many opportunities in the lab environment for pollution prevention. Good management of resources and ordering minimum quantities of toxic/hazardous materials will help the lab maintain good pollution prevention techniques.

9. WASTE MANAGEMENT

9.1 The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect air, water and land by minimizing and controlling all releases form hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and complying with all solid hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions.

9.2 It is the responsibility of the analyst to follow the safety practices as established in the safety manual and chemical hygiene plan (Sections 5 and 11) and to follow the policies and procedures established for waste disposal. Waste disposal includes but is not limited to test reagents, acids and alkaline materials, organic standards as well as contaminated samples received from clients.

9.3 It is the responsibility of the analyst or lab personnel to notify the Division Safety Officer if any substances are encountered where there are no established procedures for disposing of that substance. DO NOT dispose of samples that are of questionable nature until the proper disposal procedure has been established for that substance.

10. References

10.1. Methods for Chemical Analysis of Water and Wastes, USEPA, Environmental Monitoring and Support Laboratory EPA-600/4-79-020 Revised March 1983.

10.2. Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Updates I, II and III, Revised December 1996.

10.3. Various pre-existing internal documents (SOPs) were used as resources/references during the preparation of this document. These documents are on file at the division.

11. Method Deviations

11.1. Section 6.2 of Method 9045C requires that samples be analyzed "as soon as possible". This laboratory uses a holding time of 7 days for this analysis.

11.2. Section 7.1.2 of Method 9045C requires a minimum of 2 points for calibration. This SOP requires a minimum of 3 points for calibration.

11.3. Sections 7.2.5 and section 7.3.5 of Method 9045C require the laboratory to report results as "pH measured in water at ___°C" where "___°C" is the temperature at which the test was conducted. This laboratory does not report the temperature at which the test was conducted but does incorporate the use of a pH meter with automatic temperature correction.

Attachment A - Accumet Model 25 Calibration

1. After inserting probe in the pH 4 buffer, wait for stability indicator to change from "U" to "S" (U indicates unstable conditions and S indicates stable conditions);

2. Press the standardize button;

3. Select 1 - Update or Add Standard

4. A check list will appear reminding you of various steps that you should have already performed such as rinsing the electrode, etc. After reviewing the checklist press enter.

5. Wait for the stability indicator to change from "U" to "S" and then record the pH reading in the Calibration logbook.

6. Repeat this entire process for the pH 7 and pH 10 buffers.

Attachment B - Probes

pH Meter	Probe & Catalog #	Filling Solution

APPENDIX C

ARIZONA INSTRUMENT
USER'S MANUAL

TITLE:
JEROME 431-X MERCURY VAPOR ANALYZER OPERATION MANUAL, AZI P/N SS-086

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Approved: Blaine Nelson
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Approved: Kathy Reed
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Approved: Carolyn Gardner
Customer Service Manager

CHANGE RECORD

Rev	Date	Responsible Person	Description of Change
A	January, 1995	Carolyn Gardner	Initial Release
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C	July, 1996	Carolyn Gardner	Edit, correct and update information and graphics.

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Document Control Administrator

DOCUMENT ISSUE/CHANGE NOTIFICATION

All AZI

JEROME 431-X

MERCURY VAPOR ANALYZER

Operation Manual

July, 1996

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Part Number SS-086
Doc# 6J21-0001, Rev C

JEROME 431-X

Mercury Vapor Analyzer

Operation Manual

TABLE OF CONTENTS

FOR THOSE WHO CAN'T WAIT TO USE YOUR JEROME 431-X BEFORE READING THIS MANUAL

Remember to read the manual for added details that will optimize the results and the life of your instrument. Also refer to the manual for complete details on operation, maintenance and troubleshooting or if your application requires use of dosimeters, special voltage inputs or data output.

The Jerome 431-X is easy to operate and ready for use upon receipt from the factory. Follow these brief steps to use your instrument.

1. Remove the instrument from the packing material. Check for any damage and confirm receipt of all parts on your packing list. Contact Arizona Instrument Customer Service at 800-235-3360 if you have any questions.

2. Press the ON button. In less than one second the display should read .000. Note that a LO BATT message appears briefly in the upper left corner. (If the LO BATT light persists, it is necessary to charge the battery. See page 10 for details.)

3. Check the voltage setting (110 or 220 VAC) on the back of the instrument. Ensure that it is set to the correct voltage. (If the voltage must be changed, turn the knob. However, it may also be necessary to change the frequency setting; see page 16 for details.)

4. Perform a sensor regeneration by following these steps:

   - Plug the line cord into the instrument using the plug in the back and to an AC power outlet.
   - Power the instrument ON and press the REGEN button. The instrument will begin a 10 minute regeneration cycle, indicated by .H.H.H flashing on the display. **Do not interrupt this cycle.** (For a complete description of this process, see page 6.) If any error message, such as .H.L.P or .L.L.L appears on the display, see the Troubleshooting section on page 19.
   - Adjust the sensor zero by pressing the ZERO button and turning the zero adjust screw located under the handle. Adjust until the display reads 0.

5. The instrument is now ready to sample. Note that as the instrument measures mercury, the ZERO will display H. **Do not adjust the ZERO after the instrument has measured mercury and before the next regeneration.** (Occasionally the ZERO may drop to L (for low) between the initial zeroing and the first sample. It is OK to readjust the ZERO if the instrument has not measured mercury.)

6. The instrument is designed for work space air monitoring. Press the SAMPLE button to start a 10 second sampling cycle. **DO NOT allow the probe or the instrument's intake to come in contact with liquids. *Note that the instrument is not explosion proof.***

7. After the day's survey, again perform a sensor regeneration. When complete, store the instrument with the zero air filter in the intake.

Call AZI Customer Service, or your Technical Sales Representative, at 1-800-235-3360 or 1-602-470-1414 if you have any questions

THE JEROME 431-X
GOLD FILM MERCURY VAPOR ANALYZER

1 INTRODUCTION

The Jerome 431-X Gold Film Mercury Vapor Analyzer is designed for the easy and accurate analysis of mercury vapor in the workplace environment and for the location of mercury spills. The 431-X is easy to operate and has few maintenance requirements however, please take a moment to read this manual before attempting operation.

The Jerome 431-X is an ambient air analyzer with a range of 0.001 to 0.999 milligrams per cubic meter (mg/m^3 Hg). If you have any questions about your application or operation, please call AZI Customer Service at (800) 235-3360 for assistance.

Features of the 431-X, include:

- Automatic sensor regeneration when equipped with the communications option and used with the Jerome Communication Interface Software (JCI) program and the Jerome data logger.
- Regulated film heat voltage during sensor regeneration. This allows the sensor to clean properly with voltages from 100-130 VAC (or 200-260 VAC).
- Survey mode can be locked in.
- DIP switch setting can change the digital meter readings from mm^3 Hg to nanograms (ng) of Hg (see page 17).

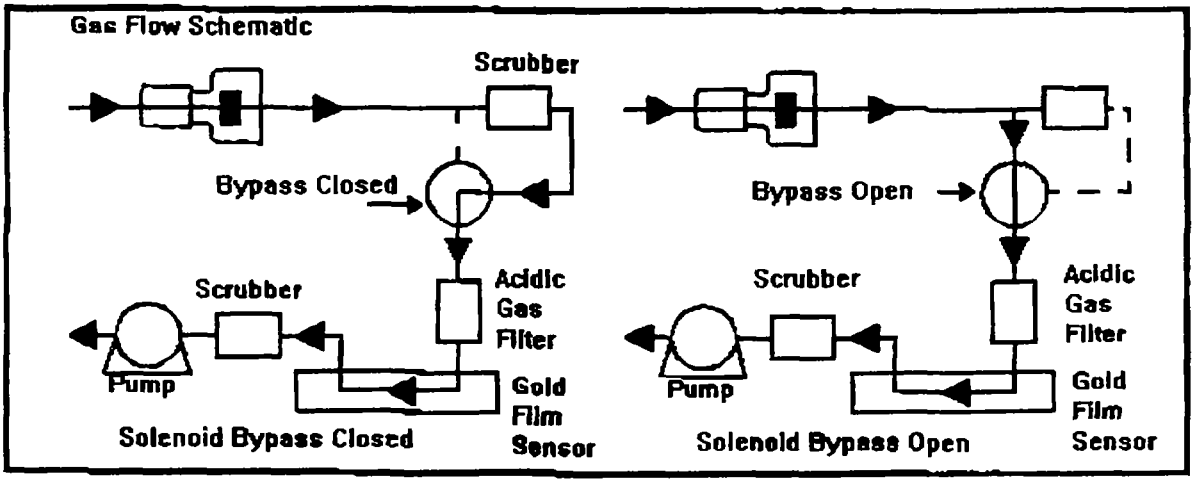
The Jerome 431-X can be operated from 100-130 or 200-260 VAC. To change the default voltage range, refer to Setting the Input Voltage, page 16.

CAUTION: The Jerome 431-X is intended for vapor use only. **DO NOT** allow the probe or the instrument's intake to come in contact with liquids, dust or other foreign material.

2 PRINCIPLE OF OPERATION

Mercury is unique in its ability to alter the resistance of a gold film. The 431-X sensor consists of two thin gold films, a reference and a sensor, configured in a Wheatstone Bridge Circuit, which detects very small changes in electrical resistance. The reference film is sealed and not exposed to mercury. The sensor film is exposed to mercury resulting in resistance changes, which are measured by the circuit. A microprocessor computes the concentration and displays the results.

Activating the SAMPLE mode starts an internal pump which draws air through a scrubber filter and into the flow system. After 2 seconds, the sample solenoid bypass opens, closing off the scrubber filter from the flow system. The sample air passes through a filter (removing any acidic gases which interfere with the sensor's response to mercury) and is drawn over the gold film sensor. The sensor adsorbs and integrates the mercury vapor. Nine seconds after starting, the sample solenoid bypass closes and the remainder of the sample is drawn through the scrubber filter and the flow system. The measured concentration is then displayed on the digital meter in milligrams per cubic meter (mg/m^3) of mercury. An internal DIP switch can be used to change the digital meter display from mg/m^3 to nanograms of mercury (see page 17).



The instrument's microprocessor automatically rezeroes the digital meter at the start of each sample cycle and retains the meter reading until the next sample cycle begins, thus eliminating drift between samples.

During the sample cycle, bars on the digital meter represent the percentage of sensor saturation. Approximately sixty-five samples containing 0.1 mg/m^3 Hg may be taken before the sensor reaches saturation. After absorbing approximately 500 nanograms of mercury, the sensor becomes saturated and needs to be cleaned. This is accomplished by a manually

activated 10 minute heat cycle, or sensor regeneration which burns the mercury from the sensor. This mercury is absorbed on internal filters to prevent any external contamination. The solenoid bypass closes during the sensor regeneration cycle, causing the air to pass through the scrubber filter, providing clean air for the regeneration process. The flow system's final scrubber prevents contamination to the atmosphere from the desorbed mercury.

After a sensor regeneration, it is necessary to bring the two gold films back to a similar resistance. The ZERO button, along with the ZERO ADJUST potentiometer, are used to reset the sensor's reference film and sensor film to the same baseline. The sensor may exhibit some low level thermal drift after the regeneration cycle, due to heat generated during sensor regeneration. To ensure maximum sample accuracy, wait 30 minutes after a regeneration and then check the ZERO adjustment. If the display reads 0 when the ZERO button is pressed, the adjustment has been accomplished. If the display reads H or L, simply turn the ZERO ADJUST pot with the trimmer tool or small screwdriver to complete the adjustment.

Only adjust the ZERO pot after a regeneration. It is not necessary to rezero between samples since the instrument automatically erases the previous reading. If the ZERO ADJUST pot is manually turned between samples, the results will be slightly lower than the actual concentration. However, this is not a permanent problem and is corrected with a sensor regeneration.

3 INSTRUMENT OPERATION

3.1 DIGITAL METER DISPLAY CODES

METER DISPLAY	EXPLANATION
000	Ready to sample
.000	Lack of mercury reading
00.0	Lack of mercury reading, display in nanograms (see page 17)
.8.8.8	Perform sensor regeneration (refer to page 6)
.H.H.H	Sensor regeneration in progress (.H.H.H flashes)
.L.L.L	Perform re-zero (refer to page 6)
.P.P.P	Power cord required or low line power, <100 VAC (or 200 VAC)(see page 17, Changing the Fuse, if .P.P.P remains on after the cord is connected.)
.H.L.P	High line power, greater than 130 VAC (or 260 VAC)
.LO BAT	Recharge batteries (refer to page 10)
.E.E.E	Same as LO BAT, automatically shuts off
.HL	*High level, sample exceeded maximum sample limit (.999)*
DURING SAMPLING	
.-	0-25% sensor saturation
.--	25-50% sensor saturation
.---	50-75% sensor saturation
-.—	75-100% sensor saturation
DURING SAMPLING	**USING THE SURVEY MODE**
-	Survey sampling (minus sign flashes continuously)
WHEN ZERO IS DEPRESSED	Adjust to 0 only after sensor regeneration. It is normal for the display to read H after sampling has started.
0	Zero, ready to sample
H	High, turn Zero pot counterclockwise
L	Low, turn Zero pot clockwise

3.2 DAILY OPERATIONS

Before each day's use of the Jerome 431-X, perform the following four steps to verify proper instrument operation:

PROCEDURE:

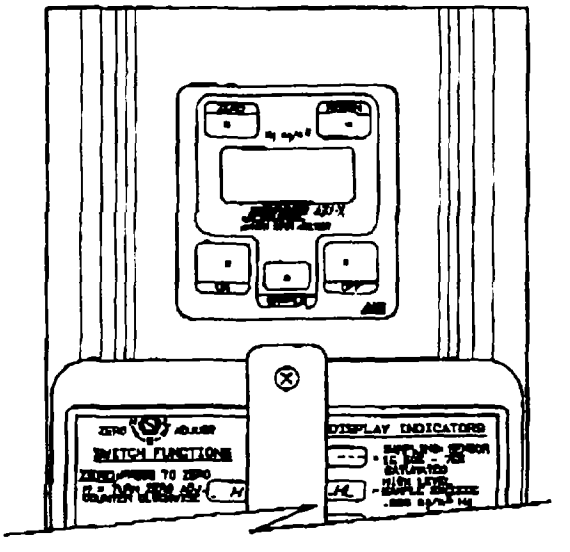
! Press the power ON button.

The digital meter displays 000. (Disregard the digital meter's initial momentary reading.) Recharge or replace the battery pack if the LO BAT indicator REMAINS ON. Refer to pages 10 and/or 15 for the procedure.

To ensure the instrument's electronics have stabilized, allow a 1 minute warm up before beginning the next step.

! Perform a sensor regeneration. Refer to page 6 for the procedure. Thirty minutes after sensor regeneration is complete, rezero the instrument.

NOTE: For maximum accuracy, such as when testing with the Functional Test Kit, wait thirty minutes after the sensor regeneration cycle to rezero the unit. For emergency response, such as for spill cleanup, the unit can be rezeroed immediately after sensor regeneration



! Press the SAMPLE button.

During the sample cycle, the digital meter displays a bar (-) which indicates the amount of sensor saturation.

! At the end of the 12 second cycle, read the digital meter.

The number shown on the digital meter is the mercury concentration in mg/m³. This value remains on the display until the next sample is taken. The digital meter automatically zeroes at the start of each sample.

! At the end of each day's use perform a sensor regeneration. **DO NOT ALLOW MERCURY CONTAMINATION TO STAY ON FILM OVERNIGHT.**

3.3 SENSOR REGENERATION PROCEDURE

A sensor regeneration is needed to clear the 431-X sensor of any accumulated mercury. This simple procedure should be done:

- At the beginning of the day on which the instrument is to be used.
- During the mercury survey, if the sensor becomes saturated.
- At the end of the day's survey, before storage.

See the Principles section on page 2 for more details on the gold film sensor and the sensor regeneration.

AC power must be between 100-130 VAC or 200-260 VAC for the sensor to clean properly. If AC power is not between these limits, an .P.P.P or .H.L.P may appear in the display (see page 4). Refer to page 16 for voltage and frequency settings.

CAUTION: Once a sensor regeneration is initiated, **DO NOT** interrupt the cycle.

PROCEDURE:

! Attach the power cord to the 431-X and plug it into AC power. AC power is required to thermally regenerate the sensor.

! Press the power ON button.

! Press the REGEN button.

The digital meter flashes .H.H.H for the duration of the 10 minute cycle and displays .0.0.0 when the cycle is completed.

DO NOT INTERRUPT THIS CYCLE. Wait until the cycle is completed before continuing with the next step.

NOTE: The digital meter will read .P.P.P after REGEN is activated if the power cord is not plugged in or if the instrument's fuse needs replacing. Plug in the power cord, or if necessary, replace the fuse according to the procedure on page 17.

! While pressing the ZERO button, turn the ZERO ADJUST potentiometer using the trimmer tool until the digital meter reads 0.

If the meter reads H, turn the ZERO ADJUST counter-clockwise;
If the meter reads L, turn the ZERO ADJUST clockwise.

See the illustration on page 5 for the location of the ZERO ADJUST potentiometer. **NOTE:** A minimum 30 minute wait after the sensor regeneration cycle is complete ensures maximum sample accuracy. The unit can be used immediately following the sensor regeneration if necessary. When the sensor regeneration is complete, press ZERO and adjust the ZERO ADJUST pot until 0 appears on the display. Install the zero air filter in the intake and take several samples or lock the instrument into survey mode (see page 9). After approximately one minute, stop sampling and check the ZERO. Adjust to 0. Repeat sampling through the zero air filter until sensor remains on 0.

NOTE: Depending upon internal configuration, a number between 00 and 100 may appear on the display, instead of H, L, or O when zero is pressed. See Internal Dip Switch Settings, page 16, for details. **IMPORTANT: Do not turn the ZERO ADJUST potentiometer between samples.** Turn the ZERO ADJUST only after a sensor regeneration cycle otherwise invalid readings will result.

! Press the power OFF button and disconnect the power cord.

! The Jerome 431-X is ready for sampling.

3.4 SAMPLE MODE

This mode, used for standard operation, produces optimum accuracy (+/- 5% at 0.100 mg/m^3 Hg) with the Jerome 431-X.

PROCEDURE:

! Press the power ON button.

The digital meter displays 000. If the unit is set to display in ng, the digital meter displays 00.0. (Disregard the digital meter's initial momentary readings.) Recharge or replace the battery pack if the LO BAT indicator REMAINS ON. Refer to pages 10 and/or 15 for the procedure.

To ensure the instrument's electronics have stabilized, allow a 1 minute warm up before beginning the next step.

! Press the SAMPLE button.

During the sampling cycle, the bar (or bars) shown on the digital display indicate the current percentage of sensor saturation. (Refer to Meter Display Codes, page 4, for code descriptions.)

NOTE: The bar (or bars) flash after 2 seconds and again after an additional 7 seconds. This flashing signals the opening and closing of the solenoid sample bypass. (See the Principles of Operation on page 2 for details.)

! At the end of the 12 second cycle, read the digital meter.

The number shown on the digital meter is the mercury concentration in mg/m³ (or ng). This value remains displayed until the next sample is taken. The digital meter automatically zeroes at the start of each sample.

When the sensor is completely saturated, the digital meter displays .8.8.8 instead of a value. No further operation is possible until a sensor regeneration is performed. (Refer to page 6 for the Sensor Regeneration procedure.)

! Press the power OFF button when not in use. Install the zero air filter in the instrument intake during storage.

SAMPLING NOTES:

The Jerome 431-X operates a minimum of 6 hours on a fully charged battery.

Use the probe (AZI P/N1400-2002) to locate mercury vapor in hard to reach places. Plug the probe directly into the instrument's intake.

CAUTION: The Jerome 431-X is intended for vapor use only. **DO NOT** allow the probe or the instrument's intake to come in contact with liquids, dust or other foreign material. Moisture or liquids drawn into the instrument can damage the sensor and flow system.

3.5 SURVEY MODE

The survey mode takes samples every 3 seconds automatically. Use this mode to locate mercury spills or to assess areas of potentially high mercury concentrations. Sampling in the survey mode is not as accurate. Due to the decreased sample volume, the accuracy of the instrument is reduced to +/- 20% @ .100 mg/m^3.

PROCEDURE:

! Press the power ON button.

The digital meter displays 000. If the unit is set to display in ng, the digital meter displays 00.0. (Disregard the digital meter's initial momentary readings.) Recharge or replace the battery pack if the LO BAT indicator REMAINS ON. Refer to pages 10 and/or 15 for the procedure.

To ensure the instrument's electronics have stabilized, allow a 1 minute warm up before beginning the next step.

! Press and **hold** the SAMPLE button.

The instrument takes a normal 12 second sample, displays the concentration at the end of the cycle and then goes into the survey mode sampling every 3 seconds. The display flashes the measured concentrations at the end of each 3 second sample cycle.

! When you are finished surveying, **release** the SAMPLE button.

The final survey value remains displayed until the next sample is taken.

NOTE: Approximately 65 samples at .1 mg/m^3 may be taken before a sensor regeneration is required.

! **To lock the instrument in a survey mode,** follow the first two steps. Hold the SAMPLE button down until the sensor status indicator bar(s) "_" begins flashing on the display. Press the ZERO button, then release the SAMPLE button. The pump should continue to run and the display should update every 3 seconds.

The instrument remains in the survey mode until one of the following occurs:

- The sensor is saturated
- A LO BAT (low battery) signal is encountered
- An HL (high mercury level) is encountered
- The instrument is turned OFF.

! Press the power OFF button when not in use.

3.6 OPERATING ON AC POWER OR GENERATOR

For stationary use, the 431-X may be operated on AC power. Operating the instrument only on AC power eliminates the need for the battery pack and its necessary maintenance. If preferred, the battery may be unplugged or removed completely.

When using a generator to power the Jerome 431-X, it is important that the generator is capable of maintaining a constant voltage output. **This is especially true during the sensor regeneration.** Use a high quality line conditioner or voltage regulator to prevent damage to the electronic components and the sensitive gold film sensor.

3.7 OPERATING ON INTERNAL BATTERY POWER

Battery power allows use of the Jerome 431-X as a portable instrument. If battery power is necessary for use, please be aware of the following:

- A fully charged battery pack (AZI P/N 2400-0907) provides power for a minimum of 6 hours of operation.
- For operating more than 6 hours, an extra fully charged battery pack is needed.
- Complete battery recharging takes 14 hours. Refer to page 10, Charging Batteries for the procedure.
- The 431-X use a rechargeable NiCad battery. Dispose of properly when replaced.
- **External battery power:** A special version of the Jerome 431-X is available that can be operated from a secondary DC source, such as a battery used in conjunction with solar panels. Contact AZI for additional information.

3.8 CHARGING BATTERIES

PROCEDURE:

- Press the power OFF button.

- Attach the power cord to the 431-X and plug it into AC power.

Complete battery recharging takes 14 hours.

The 431-X contains a trickle charger so it may be continually plugged into an AC power source without damaging the battery pack.

NOTE: To charge the batteries outside of the instrument, use the IDC Battery Charger (AZI P/N 4000-1011, for 115 VAC, P/N 4000-1012, for 230 VAC).

3.9 OBTAINING MAXIMUM BATTERY LIFE

There are certain inherent limitations to NiCad (Nickel Cadmium) batteries. The primary limitation is a memory effect that occurs when the batteries are partially discharged and then recharged, repeatedly. This memory leads to a drastic reduction in the usable battery life. To prevent this memory effect, periodically allow the battery pack to discharge completely, then recharge the battery pack.

For maximum battery life, follow these 3 steps:

- At least once a month wait until LO BAT appears on the digital meter before recharging the battery pack.
- Charge the battery pack when the LO BAT indicator comes on. Excessive discharge can damage the battery pack.
- Before storing the instrument verify the power is OFF.

When batteries fail to hold a charge, the battery pack should be replaced. Battery life under normal usage is approximately 1 year, depending on the number of charge and discharge cycles.

4 MAINTENANCE

4.1 PREVENTIVE MAINTENANCE CALENDAR

To keep the Jerome 431-X operating at peak performance, follow the maintenance schedule below. Use this schedule as a guideline only, as maintenance is more a function of application and amount of use, rather than time.

MAINTAINED PART/COMPONENT	MAINTENANCE CYCLE	REFER TO PAGE
Charge batteries	At least once per month, after 1 month's storage, or when LO BAT appears	page 10
Change .25mm fritware	Weekly or as needed	page 13
Change internal filters*	After 6 months of use or as needed.	page 14
Replace zero air filter*	Annually	page 14
Factory calibration	Annually	page 18
Calibration check	Monthly or as needed	Appendix A, page 37
Replace batteries	Annually or as needed The battery pack contains NiCad batteries. Dispose of properly.	page 15

NOTE: Install the zero air filter into the instrument's intake during storage.

*C/M filters contain Mallcosorb™, Scrubber filters and zero air filters contain Resisorb™. For safety information, see the Material Safety Data Sheets included in this manual starting on page 29. Dispose of all filters properly.

4.2 FLOW SYSTEM

The Jerome 431-X's flow system is the crucial link between the sensor and the sample. For the instrument to perform correctly, the flow system must be properly maintained. The user maintainable components of this system are the intake filter (.25 mm fritware), a C/M filter, two scrubber filters and connecting tubing.

Check the Preventive Maintenance Calendar, page 12, for a suggested schedule for changing filter disc and filters. The Tygon™ tubing in the system must be free of crimps for proper flow.

431-X Flow System

Part #	Part
Z2600-3930	Scrubber Filter
Z2600-3928	C/M Filter
Z2600-3905	Zero Air Filter
1400-3010	Tubing Adapter
2600-3039	.25mm Fritware
2500-3001	Tygon™ Tubing - 1/8" I.D. (1')
1400-3009	Intake Nozzle
PS-151	Tube Nut

4.2.1 .25MM FRITWARE

Replace the .25mm fritware once a week. In dusty environments, the fritware may need replacement as often as once a day. Replacement .25mm fritware are available from AZI, Customer Service (see Accessories & Maintenance Parts, page 27).

PROCEDURE:

! Unscrew and remove the intake from the Jerome 431-X.

! Push the old fritware disc out using your trimmer tool.

! Use tweezers to insert the new fritware.

Avoid touching the new fritware disc with fingers.

! Use the blunt end of the trimmer tool to seat the fritware disc firmly against the inner ledge of the intake.

! Screw the intake back on the Jerome 431-X.

CAUTION: The stem coming from the instrument onto which the outer intake housing is attached must be securely held in place. If loose, the tubing inside the instrument can become twisted when the intake housing is replaced. It may be necessary to open the instrument and tighten the hold-down nuts inside the instrument. Call AZI Customer Service with questions.

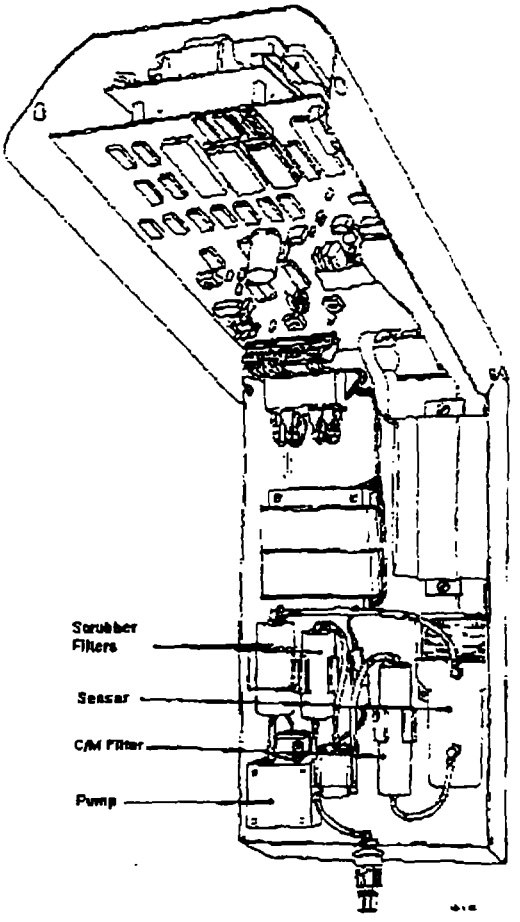
4.2.2 INTERNAL FILTERS

Replace the internal filters* (one C/M filter and two scrubber filters) after six (6) months of use, or as needed. (See Troubleshooting section, page 19.)

PROCEDURE:

- Press the power OFF button and unplug the power cord.
- Remove the 2 side screws from the intake end of the instrument and open the case.
- Carefully disconnect the Tygon™ tubing from both ends of the filters and discard the old filters.

  CAUTION: Old filters, especially the scrubber filter may contain mercury. C/M filters contain Mallcosorb™ and scrubber filters contain Resisorb™. They may contain trace amounts of mercury. For safety information see the Material Safety Data Sheets on page 29. Use proper disposal methods.



- Connect the new filters to the Tygon™ tubing, ensuring all filter nipples point toward the intake and elbows point according to the illustration.

Push the Tygon™ as far as it will go onto the filter fittings.

! Push the filters into the mounting clips.

! Remove any crimps in the tubing and ensure that tubing connections are secure.

! Close the case and replace the screws.

! Dispose of all filters in accordance with state and federal regulations.

4.2.3 REPLACING BATTERY PACK

PROCEDURE:

! Press the power OFF button.

! Unplug the power cord.

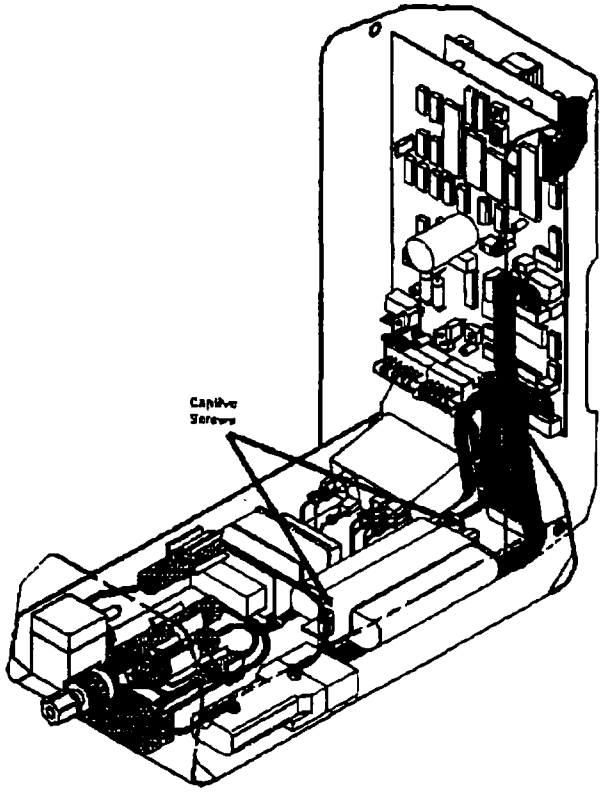
! Remove the 2 side screws from the intake end of the instrument and open the case lid.

! Disconnect the battery connector from the board.

! Loosen the 2 captive screws holding the battery bracket and remove the bracket.

! Remove the old battery pack and replace with a new battery pack.

! Replace the battery bracket and tighten the captive screws.



! Connect the new battery connector to the board.

! Close the case and replace the screws.

! Dispose of the old NiCad battery properly.

! Dispose of old nickel cadmium (NiCad) battery in accordance with state and federal regulations.

5 INTERNAL DIP SWITCH SETTINGS

5.1 SETTING THE INPUT VOLTAGE

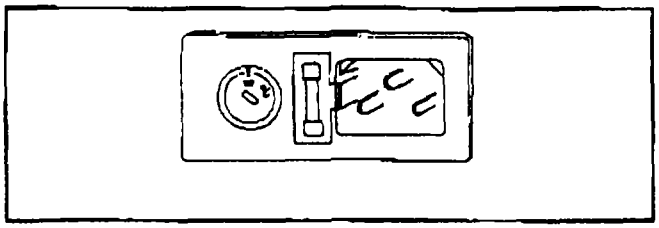
This instrument has been factory set and calibrated to use the requested power setting (either 110 VAC or 220 VAC). The voltage setting is, however, easily changed to use either 110 VAC or 220 VAC.

PROCEDURE:

! Ensure the instrument is turned OFF and unplugged.

! Locate the power receptacle on the rear of the instrument.

! Insert a small screwdriver in the voltage selection slot and turn the selector until the arrow points toward your setting choice and a click is heard.



5.2 SETTING THE INPUT CYCLES

The 431-X has been set to the desired electric cycle, either 50 or 60Hz. Proper cycle setting is necessary to ensure a proper sensor regeneration.

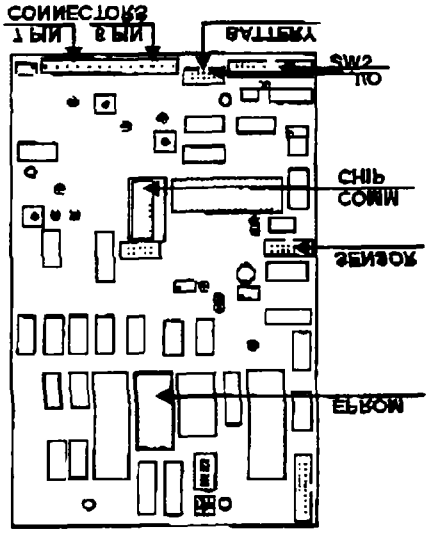
PROCEDURE:

! Open the instrument lid.

! Locate SW2 at the top of the main circuit board (see figure, page 14)

! Set the SW2 switch to the appropriate cycle (Hz).

	60Hz	50Hz
SW#2		
Dip switch #1	OFF	OFF
Dip switch #6	OFF	ON



5.3 DISPLAYING NANOGRAMS OR MILLIGRAMS/CUBIC METER

The instrument is factory set to display mg/m³ (milligrams per cubic meter) Hg (.XXX). For some applications, including dosimeter analysis, the instrument's display can be converted to display nanograms.

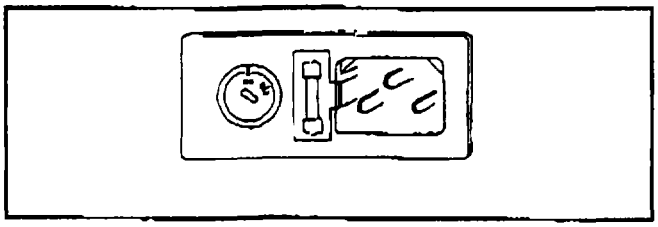
PROCEDURE:

! Turn the instrument off. Remove the two screws near the front of the instrument and open the lid.

! Locate SW2 (see diagram, page 16).

! Switch dip switch # 2 to OFF for nanogram display.

5.4 CHANGING THE FUSE



If the instrument display reads .P.P.P when the instrument is connected to AC power or when REGEN is pressed, or if the battery will not charge, the fuse may need to be replaced. The AC line power could also be less than 100 VAC (220 VAC). Check the fuse or the AC line power with a voltage meter.

Fuse Replacement:

PROCEDURE:

! Locate the power receptacle on the rear of the instrument.

! Insert a small screwdriver in the slot (see figure above) and gently slide the fuse compartment out.

! Remove and discard the fuse held in the open sided clip and replace it with the spare fuse held in the boxed spare fuse compartment.

! Replace the fuse compartment in the power receptacle.

! Replace the spare fuse with another 1A 250V Fast-Blo fuse (AZI P/N 5100-1012).

6 CALIBRATION

The Jerome 431-X's gold film sensor is inherently stable and does not require frequent calibration. The interval between calibrations depends upon the application and frequency of use; however, the recommended minimum or maximum interval is every 12 months.

The Jerome 431-X has been factory calibrated using NIST traceable permeation tubes. These permeation tubes have a rated accuracy of +/- 2%. In order to calibrate the Jerome 431-X, a sophisticated calibration system is required that ensures stability of the calibration gas source, eliminates any pressure in the calibration gas stream and controls the temperature of the calibration environment. Calibration also requires special proprietary software.

We strongly recommend you take advantage of our calibration and maintenance service at Arizona Instrument. Call Customer Service at 800-235-3360 OR 602-470-1414 to arrange re-calibration. A certificate of calibration is issued from AZI when your instrument is factory calibrated.

6.1 VERIFICATION OF CALIBRATION AND QUALITY CONTROL

The Functional Test Kit (AZI P/N 4431-0902) is used to determine if your instrument is within calibration tolerances between recommended annual factory calibrations. It allows you to have complete confidence in the sample results. This test verifies proper instrument operation through the introduction of a known mass of mercury into the Jerome analyzer.

If your application requires frequent verification of instrument function, this test demonstrates the unit's operation, calibration, and function. Recording Functional Test Kit results in an instrument log provides a quality control/quality assurance record of instrument function between regular calibrations. If test results fall within the expected range, you may assume the instrument is functioning correctly. **THIS TEST DOES NOT CALIBRATE THE INSTRUMENT.**

See page 37 in Appendix A, for complete Functional Test Kit procedures.

To order the kit, contact AZI Customer Service at 800-235-3360 or 602-470-1414.

7 431-X TROUBLESHOOTING

Symptom	Possible Cause	Solution
[redacted]		
Unit does not turn ON. LCD displays 000 when connected to power cord and ON button is pressed.	Dead battery	Recharge battery (minimum 14 hours) refer to page 10. Replace battery, refer to page 15.
Unit does not turn on when connected to AC power cord.	Fuse	Replace fuse, refer to page 17 .
	Insufficient power	Be sure there is power to the AC outlet using a volt meter.
[redacted]		
LCD displays .8.8.8.	Sensor saturated	Do not attempt to rezero. Unit must be regenerated. See page 6 for information.
LCD displays .L.L.L when taking first sample.	Changes in temperature	Readjust zero pot. See page 5 for information .
LCD displays H at finish of sensor regeneration	Internal contamination may redeposit mercury from flow system onto gold film sensor.	Remove and replace intake filter disk, Tygon™ tubing and internal C/M filter. Check tubing for kinks or crimps. Repeat regeneration cycle. See page 6 for information.

Symptom	Possible Cause	Solution
Zero adjust pot cannot be adjusted to 0	Pot not turned sufficiently	Turn zero adjust up to 20 times to reach the end. Pot will "click" softly.
Display still unchanged	Sensor may be ruptured or pot may be broken	Turn pot slowly in opposite direction till display reads 0. If still unchanged, call AZI Customer Service.
Sample Problems		
Air flow is restricted during the sensor regeneration cycle, causing possible permanent damage.	Kinks and crimps in the Tygon™ tubing.	Periodically check the Tygon™ tubing inside the instrument.
High erratic results	Internal mercury contamination	1. Install zero air filter in intake and tighten intake nut. Press SAMPLE button. After 3 samples, if readings are over .003 mg/m^3, replace intake filters and Tygon tubing. 2. Perform a REGEN with zero air filter in intake. See page 6 for information. Retest if necessary. Replace intake filters and Tygon™ tubing.

Symptom	Possible Cause	Solution
High/erratic results Readings vary more than 0.05 when in survey mode.	Film connection	Press and hold SAMPLE button for 12 seconds in clean area or with zero air filter in intake. Move unit from side to side, or up and down during sample cycle. Call AZI Customer Service.
Low response or erratic readings after a long period of non-use	May need a second regeneration cycle.	Wait 20 minutes between regeneration cycles. Test with FTK. See page 37 for information. If still unresponsive, call AZI Customer Service.
False readings, may go to .8.8.8 or .L.L.L	Extremely cold or extremely warm air sampled into unit	If sampling under these conditions, install zero air filter in intake. Sample until display reads .003 mg/m^3 or less. This equilibrates sensor temperature with the temperature of the sample air stream. Remove filter and take samples.
High/erratic results	Intake and internal filters may get clogged and need replacement when sampling in a dusty area	Open instrument to check for pinched, crimped or disconnected internal tubing. In extreme conditions a particle filter may be installed on intake.

Symptom	Possible Cause	Solution
Display reads .P.P.P when regeneration is attempted.	Power cord not attached	Check power cord for connection
	Blown fuse	Replace fuse. See page 17 for information.
	Line voltage less than 100 VAC (or less than 200 VAC for 220 unit)	Check line voltage settings. See page 16 for information.
	Cycles dipswitch (50 or 60Hz) set incorrectly	Check input cycle settings. See page 16 for information..
		If fuse and line voltage are OK, it may be circuit board adjustment or component, call AZI Customer Service for information.
Display reads .E.E.E	Very low battery	Recharge battery. See page 10 for information.
If battery is charged		Replace battery. See page 15 for information..
	Blown fuse	Replace fuse. See page 17 for information.
	Internal component failure	Call AZI Customer Service for information.

8 JEROME 431-X TECHNICAL SPECIFICATIONS

Range	0.003 to 0.999 mg/m^3
Sensitivity	0.003 mg/m^3 Hg
Precision	5% relative standard deviation @ 0.100 mg/m^3 Hg
Accuracy	+/- 5% @ 0.100 mg/m^3 Hg
Response time-sample mode	12 seconds
Response time-survey mode	3 seconds
Flow rate	750cc/min (0.75 liters/min)
Power requirements	100-130 VAC (or 200-260 VAC) 115 watts maximum
Batteries	Rechargeable Nickel Cadmium
Fuse	1A 250V 5 X 20 Fast Blo
Construction	Aluminum alloy
Dimensions	15 cm x 33 cm x 10 cm (6" w x 13" l x 4" h)
Weight	3.18 kilos (7 pounds)
Digital meter	Liquid crystal display (LCD)
Operating environment	0° - 40°C, non-condensing, non-explosive

8.1 Optional "Communications" Version

Alarm output	30V DC, 100mA
Dosimeter power output	For dosimeter analysis
Data output	1. Digital, Serial, RS232, Baud Rate 1200 for use with Data Logger, Base Station, and/or JCI program software 2. Digital, Serial, RS232, data format, but with driver for 20mA capability and 0 & 20mA logic levels; Baud Rate 1200 (special industrial applications)

"OPTION BOARD"

Data output	0 - 2V or 4 - 20 mA
Auto sample interval	5, 15, 30, 60 minutes
Auto regeneration interval	6, 24 or 72 hours

8.2 Instrument I/O Interface

The 431-X I/0 port (25 pin D-sub) has six functions:

- Serial communication channel, RS-232
  Interface type RS-232C full duplex, DCE
  Communication parameters - 1200 Baud, 1 start bit, 8 data bits, 2 stop bits, no parity
  Pin assignments:

Pin 1	Protective ground
Pin 2	Data in
Pin 3	Data out
Pin 7	Data ground

- Serial communication channel, 20mA current loop
  Interface type: 20mA current loop, full duplex
  Communication parameters - 1200 Baud, 1 start bit, 8 data bits, 2 stop bits, no parity
  Pin assignments:

Pin 1	Protective ground
Pin 4	Data out (+)
Pin 5	Data in (+)
Pin 14	Data out (-)
Pin 16	Data in (-)

! Alarm output

Maximum voltage	30 VDC
Maximum current	100mAmp

Pin assignments:

Pin 9	Switched battery+
Pin 10	Alarm output (open collector, active low)
Pin 7	Battery ground
Pin 23	Battery ground

! Dosimeter power

Voltage 24 - 28 volts AC

Pin assignments:

Pin 22	Dosimeter enable
Pin 23	Battery ground
Pin 12 & 24 Tied together	Dosimeter power
Pin 13 & 25 Tied together	Dosimeter power

Connecting pin 22 to 23 enables the dosimeter desorption cycle.

! Switched battery connection for data logger

Pin assignments:

Pin 9	Battery +
Pin 7	Battery ground
Pin 23	Battery ground

! Unswitched battery connection for external battery pack pin assignments

Pin assignments:

Pin 15	Battery +
Pin 19	Battery +
Pin 7	Battery ground
Pin 23	Battery ground

NOTE: Pins 6, 8, 11, 17, 18, 20 and 21 are non-standard and should not be connected.

8.3 POTENTIAL INTERFERENCES

Potential interferences to the Jerome mercury vapor analyzers are rare and most of these can be eliminated with proper maintenance procedures. However, erroneously high readings can sometimes occur. Here are a few things to be aware of when using the instruments.

The gold film sensors used in the Jerome mercury vapor analyzers do not respond to the following compounds:

- Hydrocarbons
- CO, CO_2, and SO_2
- Water vapor (Note that water vapor condensation on the gold film can cause irreparable harm to the sensor and must be avoided.)

The acidic gas filter, contained in the internal filter system, removes the following compounds that cause the gold film sensor to respond:

- Chlorine
- NO_2
- Hydrogen Sulfide (H_2S)
- Most mercaptans (organic sulfur compounds or "thiols")

In areas containing these highly volatile compounds, the filter can become quickly saturated. In such situations, it is recommended that these gases be allowed to dissipate before sampling for the less volatile, more persistent mercury vapor. Collection of air samples with Jerome gold coil dosimeters for analysis by the Jerome mercury vapor analyzers will also eliminate interferences.

Ammonia in very high concentrations can cause an offgassing of accumulated acidic fumes from the internal acidic gas filter, resulting in positive readings on the instrument. In these cases, the ammonia odors are very strong. Again, either allow the vapors to dissipate or use the dosimeters. Filter replacement at regular intervals, or when unexpectedly high readings are encountered in areas of these potential interferents, may resolve these problems.

Volatile mercury compounds in general will cause the gold film to respond. Alkyl organic mercuries such as methyl mercury (and other "straight chained" compounds) are typically extremely volatile and change the electrical resistance of the gold film sensor. Any such responses should be considered "qualitative," **not** quantitative. The instruments are designed and calibrated to elemental mercury vapor only.

Inorganic mercury salts such as mercuric chloride are not very volatile. They may, however, generate some minute level of elemental mercury vapor to which the instruments will respond. This response, again, should be considered a qualitative response only.

9 ACCESSORIES & MAINTENANCE PARTS

PART #	ITEM DESCRIPTION
Y431-0901	**431 Accessory Kit** Includes: zero air filter (1), .25mm fritware (20), trimmer tool (1), probe (1) & tubing adapter (1)
Y431-0902	**Functional Test Kit** Includes: calibration vessel (1), stopper assembly (1), vial with Hg (1), syringe assembly (1), disposable syringe needles (5), septum holder assembly opt. (1) & septa - 1/4" (20)
Y431-0903	**431 Maintenance Kit** Includes: zero air filter (1), .25 mm fritware (60), 021 battery pack (1), C/M filter (1), scrubber filters (2) & 1 foot Tygon™ tubing - 1/8" I.D.
Y411-0904	**031/411 Carrying Case Assembly** Includes: case & die cut foam rubber Holds: Jerome 431-X, personal mercury dosimeters & accessories
1400-0052	**001 Field Carrying Case Assembly**
Y431-0905	**Standard Dosimeter Analysis Kit** Includes: dosimeter lead set (1), low flow pump - 2cc/min (1), personal mercury dosimeters (2), zero air filter (1), 1/8" - 1/16" adapter (1), 2' Tygon™ tubing - 1/8" I.D., 1' Tygon™ tubing - 1/16" I.D.
X412-0901	**Personal Mercury Dosimeter**
2100-6017	**Dosimeter Lead Set**
2600-2011	**Dosimeter Pump - 2cc/min**
Z2600-3911	**10:1 Dilution Module Assembly**

9.1 431-X Flow System

Z2600-3930	Scrubber Filter
Z2600-3928	C/M Filter
Z2600-3905	Zero Air Filter
1400-3010	Tubing Adapter
2600-3039	.25mm Fritware
2500-3001	Tygon™ Tubing - 1/8" I.D. (1 foot)
1400-3009	Intake Nozzle
PS-151	Tube Nut

9.2 Replacement Parts

Z4000-0907	021 Battery Pack Assembly
4000-1011	IDC Battery Charger - 115 VAC
4000-1012	IDC Battery Charger - 230 VAC
1300-0025	1/8" - 1/16" Tubing Adapter
1400-2002	Probe
2300-0001	Trimmer Tool
2500-3002	Tygon™ Tubing - 1/16" I.D. (1 foot)
6000-4003	Line Cord
5100-1012	Fuse 1 AMP 5 X 20 250V Fast-Blo

9.3 Test Equipment

2600-0030	Calibration Vessel
A2600-0902	Stopper Assembly Includes: rubber stopper, thermometer & needle guide
A2600-0903	Syringe Assembly Includes: syringe, syringe holder & needle
2600-0022	Syringe Needles (2)
Z2600-3914	Septum Holder Assembly
3200-0011	Septum - 1/4" (20)
A2600-0904	Vial with Hg

For current prices and delivery information, call AZI Customer Service at 800-235-3360 or 602-470-1414.

FACTORY CALIBRATION SERVICE
Service includes filter replacement, component check and calibration and instrument calibration to NIST traceable standards.

For authorization & scheduling, call AZI Customer Service at 800-235-3360 or 602-470-1414.

10 MATERIAL SAFETY DATA SHEET

Date of Issue 04/95

10.1 MALLCOSORB

Arizona Instrument Corporation
4114 East Wood Street
Phoenix, AZ 85040
INFORMATION HOTLINE (800) 235-3360

Product Identification
SYNONYMS: Soda lime solid; sodium hydroxide mixed with lime
FORMULA CAS NO.: 8006-28-28
MOLECULAR WEIGHT: N/A
HAZARDOUS INGREDIENTS: N/A
CHEMICAL FORMULA: N/A

Section 1 - Physical Data
APPEARANCE: White deliquescent pellets
ODOR: Odorless
BOILING POINT: No information found
MELTING POINT: No information found
VAPOR PRESSURE @ 20°C: Essentially zero
SPECIFIC GRAVITY: No information found

Section 2 - Fire and Explosion Data
FIRE: Not combustible, contact with moisture may generate heat to ignite combustibles.
EXPLOSION: Possible when in contact with incompatible materials.
FIRE HAZARD: Full protective clothing & NIOSH approved self-contained breathing apparatus.

Section 3 - Reactivity Data
STABILITY: Causes no hazardous decomposition or hazardous polymerization
INCOMPATIBILITIES: Water, steam, acids, fluorine & many organics; contact with nitro compounds cause formation of flammable hydrogen gas.

Section 4 - Leak/Spill Disposal Information
PRODUCT CLEAN-UP: Protective clothing & respiratory protection, scoop up spilled material, avoid dusting, neutralize traces with dilute acid.
DISPOSAL: Transfer to closed metal container & dispose of according to local, state & federal regulations. DO NOT CONTACT WITH WATER.

Section 5 - Health Hazard Information
OSHA PERMISSIBLE EXPOSURE LIMIT(PEL):
Calcium Oxide 5 mg/m^3 (TWA)
Sodium Hydroxide 2 mg/m^3 (TWA)
ACGIH THRESHOLD LIMIT VALUE (TLV):
Sodium Hydroxide 2 mg/m^3 (TWA)
Calcium Oxide 2 mg/m^3 (TWA)

EXPOSURE/HEALTH EFFECTS:
INHALATION - Upper respiratory tract damage, pneumonitis;
INGESTION - Severe mouth, throat & stomach burns, severe tissue scarring & death may result;
SKIN & EYES - Irritation or severe burns, possible blindness resulting
FIRST AID:
INHALATION - Remove to fresh air; if not breathing, give artificial respiration; if breathing is difficult, give oxygen; get medical attention immediately.
INGESTION - DO NOT INDUCE VOMITING! give large quantities of water or milk; get medical attention immediately.
SKIN & EYES - Immediately flush with water for 15 minute minimum; remove contaminated clothing.

Section 6 - Special Protection Information
Ventilation must be sufficient to meet TLV. Wear rubber gloves & eye protection.

Section 7- Storage and Special Information
Keep in tightly closed container, in cool, dry ventilated area, away form incompatible substances.

MALLCOSORB

Addendum to Material Safety Data Sheet

ARIZONA INSTRUMENT CORPORATION
4114 East Wood Street
Phoenix, AZ 85040
INFORMATION HOTLINE (800) 235-3360

Hazard Categories for SARA Section 311/312 Reporting

Acute	Chronic	Fire	Pressure	Reactive
X				

Product or Components of Product	SARA EHS Sect. 302		SARA 313 Chemicals Sect.		CERCLA Sec103 RQ (lbs)	RCRA Sec. 261.33
	RQ (lbs)	TPQ (lbs)	Name List	Chemical Category		
MALLCOSORB™ Sodium hydroxide (1310-73-2) 1-10%	No	No	Yes	No	1000	No
Calcium chloride (10043-52-4)	No	No	No	No	No	No
Ethyl violet (2390-59-2)	No	No	No	No	No	No
Calcium hydroxide (1305-62-0)	No	No	No	No	No	No
Actual concentrations proprietary						

SARA Section 302 EHS RQ: Reportable quantity of extremely hazardous substance, listed at 40 CFR 355.
SARA Section 302 EHS TPQ: Threshold-Planning Quantity of extremely hazardous substance. An asterisk (*) following a Threshold Planning Quantity signifies that if the material is a solid and has a particle size equal to or larger than 100 micrometers, the Threshold Planning Quantity = 10,000 lbs.
Section 313 Chemicals: Toxic substances subject to annual release reporting requirements listed at 40 CFR 372.65.
CERCLA Sec. 103: Comprehensive Environmental Response Compensation and Liability Act (Superfund) Releases to air, land or water of these hazardous substances which exceed the Reportable Quantity (RQ) must be reported to the National Response Center (800) 414-8802; listed at 40 CFR 302.4.
RCRA: Resource Conservation and Reclamation Act. Commercial chemical product wastes designated as acute hazards and toxic under 40 CFR 261.33

MATERIAL SAFETY DATA SHEET

Date of Issue 04/95

10.2 MERCURY

ARIZONA INSTRUMENT CORPORATION
4114 East Wood Street
Phoenix, AZ 85040
INFORMATION HOTLINE (800) 235-3360

Product Identification:
CHEMICAL NAME: Mercury metal
TRADE NAME & SYNONYMS: Quick Silver
CHEMICAL FAMILY: Metals
FORMULA: Hg
FORMULA WEIGHT: 200.59

Section 1 - Physical Data
ODOR: Odorless
SPECIFIC GRAVITY (H_2O = 1): 13.54
VAPOR PRESSURE AT 20°C (mmHg): 0.0012
BOILING POINT, 760 mm Hg (°C): 356.9
MELTING POINT (°C): -38.9

Section 2 - Fire and Explosion Data
FIRE HAZARD: Nonflammable
UNUSUAL HAZARDS: Extremely toxic vapors upon exposure to high temperatures.

Section 3 - Reactivity Data
STABILITY: Stable at room temperature
INCOMPATIBILITIES AND REACTIVITIES: Acetylene, ammonia, chlorine dioxide, azides, calcium (amalgam formation), sodium carbide, lithium, rubidium, copper, nitric acid

Section 4 - *Leak/Spill* Disposal Information
PRODUCT CLEAN-UP: Recover with suction cup equipped with a capillary tube.
DISPOSAL METHOD: Perform in compliance with all current local, state and federal regulations.

Section 5 - Health Hazard Information
EXPOSURE LIMIT
0.05mg/m³ (NIOSH/TWA)
0.100mg/m³ Ceiling (OSHA)

EXPOSURE/HEALTH EFFECTS: Coughing, bronchitis, pneumonia, tremor, insomnia, irritability, headache, fatigue, weakness, stomatitis, weight loss, GI disorder
SKIN & EYES: Can irritate skin and eyes
FIRST AID:
SKIN: Wash with water, get medical assistance.
EYES: Wash with water, get medical assistance.
INHALATION: Remove to fresh air, get medical assistance.
INGESTION: Get medical assistance.

Section 6 - Special Protection Information
Ventilation must be sufficient to meet TLV. Wear rubber gloves and eye protection.

Section 7 - Special Handling and Storing Precautions
Do NOT heat mercury unless appropriate safety precautions for highly toxic vapors have been take. Store in sealed container.

Section 8 - Hazardous Ingredients
Mercury and Mercury vapor

MATERIAL SAFETY DATA SHEET

Date of Issue 04/95

10.3 RESISORB

ARIZONA INSTRUMENT CORPORATION
4114 East Wood Street
Phoenix, AZ 85040
INFORMATION HOTLINE (800) 235-3360

Product Identification:
PRODUCT NAME: Resisorb - Mercury Vapor Absorbent
FORMULA CAS NO.: 00000-00-0
MOLECULAR WEIGHT: .00
CHEMICAL FORMULA: Proprietary mixture

Section 1 - Physical Data
APPEARANCE & ODOR: Black solid with halogen-like odor
BOILING POINT: N/A
MELTING POINT: N/A
VAPOR PRESSURE: N/A
SPECIFIC GRAVITY: N/A

Section 2 - Fire and Explosion Hazard Data
FIRE: Combustible, keep away from heat, sparks, flame.
EXPLOSION: Contact with strong oxidizers may cause explosion.
FIRE HAZARD: Use water spray to soak, class A extinguisher, gull protective clothing & NIOSH approved self-contained breathing apparatus, move exposed containers from fire area if it can be done without risk, if not, use water to keep fire-exposed containers cool.

Section 3 - Reactivity Data
STABILITY: Stable, no hazardous polymerization
CONDITIONS TO AVOID: Heat, flame, sources of ignition
INCOMPATIBILITIES: Strong oxidizing agents, nitric acid, ammonia, alkali metals, strong reducing agents

Section 4 - Leak/Spill Disposal Information
PRODUCT CLEAN-UP: Protective clothing & respiratory protection, scoop up spilled material, avoid dusting, flush spill area with water.
DISPOSAL: Transfer to clean, dry container & dispose of in accordance with local, state & federal environmental regulations.

Section 5 - Health Hazard Information

EXPOSURE/HEALTH EFFECTS:
INHALATION: May cause tightness & chest pain, coughing & difficulty in breathing.
INGESTION: May cause nausea, vomiting, headaches.
SKIN AND EYES: Dust may irritate skin and/or eyes.

FIRST AID:
INGESTION: get medical attention, if conscious, immediately induce vomiting.
SKIN AND EYES: Immediately flush with water for 15 minute minimum; remove contaminated clothing.

Section 6 - Special Protection Information
Use adequate general or local ventilation to keep fume or dust levels as low as possible. If airborne concentration is high, use respirator or dust mask. Wear rubber gloves & eye protection.

Section 7 - Storage and Special Information
Keep in tightly closed container, in cool, dry ventilated area, away from heat, sparks or flame; isolate from incompatible substances.

MATERIAL SAFETY DATA SHEET

10.4 NICKEL CADMIUM BATTERY

MATSUSHITA BATTERY INDUSTRIAL CO
1 Matsushita-Cho
Moriguchi Osaka 570 JAPAN
EMERGENCY TELEPHONE 201-392-6703
INFORMATION TELEPHONE 714-373-7538

Product Identification:
PRODUCT NAME: Nickel Cadmium Battery
HAZARDOUS INGREDIENTS: $Ni(OH)^2$, NiOOH, Cd, $Cd(OH)^2$, KOH or NaOH, LiOH
CHEMICAL FORMULA: NiCd

Section 1 - Physical Data
APPEARANCE & ODOR: None
BOILING POINT: Approximately 170°C
MELTING POINT: N/A
VAPOR PRESSURE: N/A
SPECIFIC GRAVITY: 2.6

Section 2 - Fire and Explosion Data
FIRE HAZARD: Under normal charging and discharging, no fire hazard exists.
EXPLOSION: Under normal charging and discharging, no explosion hazard exists.

Section 3 - Reactivity Data
STABILITY: Extremely stable
INCOMPATIBILITIES: N/A

Section 4 - Leak/Spill Disposal Information
PRODUCT CLEAN-UP: Non-toxic in normal use
DISPOSAL METHOD: DO NOT incinerate. Dispose of in discharged state to avoid shorting.

Section 5 - Health Hazard Information

EXPOSURE/HEALTH EFFECTS:
INHALATION: N/A
INGESTION: N/A
SKIN AND EYES: May irritate if contact is made with the electrolyte (alkaline).

FIRST AID:
INHALATION: N/A
INGESTION: N/A
SKIN AND EYES: Immediately flush affected area with cool water. If contact is made with the eyes or mucous membranes, immediately flush with water and get medical assistance.

Section 6 - Special Protection Information
No special protection required in normal usage.

Section 7 - Storing and Special Information
No special precautions required for storing.

11 APPENDIX A - 431-X FUNCTIONAL TEST

If your application requires frequent verification of instrument functionality, this test will benefit you. If the test results fall within the expected range, you may assume the instrument is functioning properly. This test does not calibrate the instrument.

NOTE: Perform the functional test ONLY after a sensor regeneration.

The 431-X Functional Test Kit contains all accessories necessary to perform the functional test:

- calibration vessel (1)
- stopper/thermometer assembly (1)
- vial of Hg* (1)
- syringe assembly (1)
- syringe needles (5)
- septum holder assembly (1)
- septa (20)

CAUTION: The vial and thermometer contain liquid mercury and are possible sources of mercury contamination. Follow the instructions carefully.

***For information on special protection and health hazards, READ the Mercury Material Safety Data Sheets (MSDS), page 31 before handling or transferring the mercury into the Functional Test Kit Vessel.**

11.1 PREPARATION

! Carefully unpack and inspect the parts of the kit.

ENSURE that the mercury shipping container and mercury filled thermometer are not broken.

VERIFY that all the parts to the kit are present.

! In a ventilated area, preferably under a fume hood, remove the mercury vial from its shipping container.

! Place the functional test kit vessel and the mercury vial close to each other and open the mercury vial.

CAUTION: The edge between the plastic case and the glass inner vessel of the functional test kit vessel are not sealed well enough to prevent mercury from entering the area between the inner and outer vessels. **ENSURE** the mercury, handled in the next step, does not come in contact with the seal where the glass and plastic portions join.

NOTE: The vessel may be disassembled to transfer the mercury and better prevent contamination of the outer portion of the vessel. Instructions to disassemble the vessel can be found on page 36.

11.2 MERCURY TRANSFER

PROCEDURE:

! CAREFULLY pour the mercury into the center of the functional test kits vessel's opening.

! ENSURE that no mercury residue is on the outside of the vessel. If mercury residue is present, see the mercury Material Safety Data Sheet (MSDS) on page 31 for clean-up instructions.

! INSTALL the stopper assembly into the functional test kit vessel carefully, to prevent breakage of the thermometer.

PRESS the stopper assembly into the vessel to achieve a good seal.

! USE the 431-X instrument to verify that the outside of the vessel is not contaminated and the mercury vapor emission level, if any, is below the OSHA TLV for mercury.

! ALLOW the kit to adjust to room temperature for at least two (2) hours before using.

The temperature range for the test is 18-22°C. Avoid temperature fluctuations.

CAUTION: Do not use the calibration vessel as a portable container. If the calibration vessel is upset or greatly agitated, mercury droplets will cling to the thermometer stem, the rubber stopper, the mouth of the calibration vessel and the needle guide.

11.3 VESSEL DISASSEMBLY

CAUTION: The inner portion of the vessel is made of glass. Handle the vessel carefully to prevent breakage.

- LOOSEN, BUT DO NOT REMOVE the base of the vessel. The base unscrews from the body.
- SET the vessel on a firm surface.
- HOLD the base stationary and unscrew the body from the base.
- HOLD the base and the inner glass vessel with one hand while removing the body and gasket with the other hand.
- After the mercury is transferred into the glass inner vessel, reassemble in the reverse order.

11.4 REPLACING MERCURY

An oxide coating will form on the drop of mercury and will cause lower readings in your testing. Gently swirl the vessel to disturb the outer oxidized surface of the droplet. If this does not restore higher readings, it may be necessary to replace the mercury.

PROCEDURE:

! Carefully remove the stopper assembly from the calibration vessel.

BE SURE NEEDLE GUIDE IS FREE OF LIQUID MERCURY.

! Carefully pour the mercury into a disposal vessel. Refer to Vessel Disassembly Instructions on 36.

Mercury can become trapped between the plastic calibration vessel and the glass inner-liner.

! Replace the oxidized mercury with approximately ½ cc fresh mercury. (AZI P/N A2600-0904)

Do NOT use the syringe for measuring liquid mercury. Dispose of oxidized mercury properly.

! Reassemble the calibration vessel.

! Reinstall the stopper assembly.

11.5 FUNCTIONAL TEST PROCEDURE

NOTE: Perform the functional test **ONLY** after a sensor regeneration.

PROCEDURE:

! Leave the calibration vessel at stable room temperature for at least 2 hours.

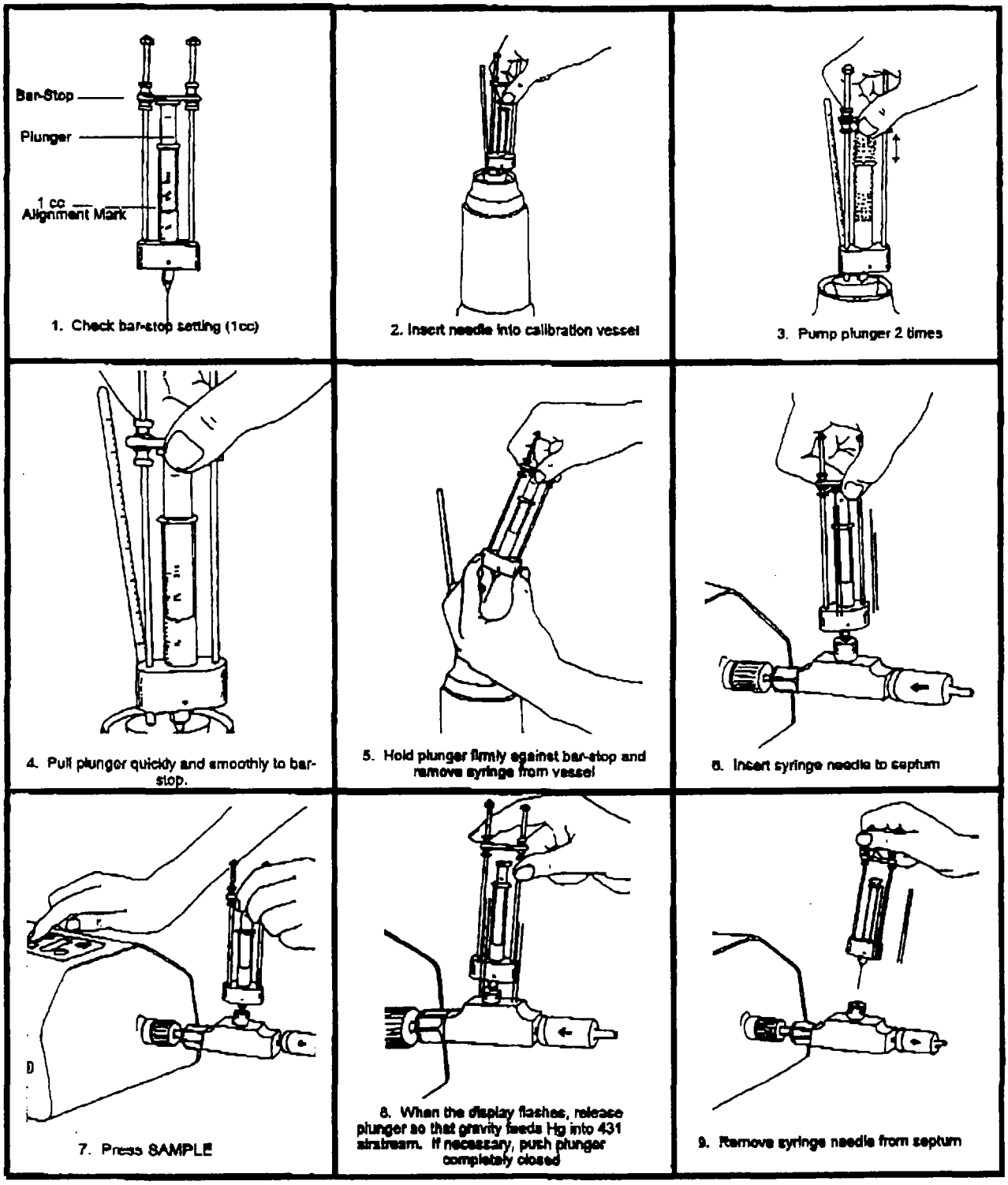
The temperature range for the test is 18° - 22°C.

Temperature fluctuations during the test procedure will produce erratic results.

! Replace the .25mm fritware.

Refer to page 13 of the 431-X manual.

11.6 SYRINGE TECHNIQUE



! Replace the septum on the septum holder assembly.

! Plug the tubing adapter end of the septum assembly into the instrument's intake and tighten the intake tube nut.

NOTE: To check for a tight seal, gently pull on the septum holder assembly. If it comes out of the intake, it may be necessary to remove the intake tube from the instrument and firmly press the tubing adapter through the intake. Tighten the intake tube firmly to the intake stem.

! Attach a zero air filter to the septum assembly.

! Press power ON.

! Take 3 samples.

If the average meter reading is less than .005, continue to the next step.

If the average meter reading is greater than .005, stop here. The instrument may be contaminated. See the Troubleshooting section, page 19.

! Note the temperature of the calibration vessel.

! Press the SAMPLE button, wait 2 seconds and **when the display flashes**, inject 1 cc of mercury vapor according to the syringe technique described on page 38. Be sure all mercury vapor has been injected before the solenoid closes (second click and display flash).

NOTE: To minimize error, it is important to carefully follow this procedure.

! Record the meter reading.

! Repeat the last two steps three times.

The last three 1cc injections should be within +/- 5% of each other. If not, refer to page 38 for proper syringe technique and repeat the procedure.

! Refer to the Temperature Conversion Chart, page 40, for the acceptable range.

The average of the last three digital meter readings should fall within the range shown on the chart.

IF THE AVERAGE IS WITHIN RANGE, THE JEROME 431-X IS FUNCTIONING PROPERLY.

- Perform a sensor regeneration. Press ZERO and turn the ZERO ADJUST (refer to page 6 in the 431-X manual for the complete sensor regeneration procedure).
- Wait 1 hour before proceeding to the next step.
- Repeat this test procedure.

If the average of the digital meter readings is still not within range, refer to page 41 Functional Test Troubleshooting.

431-X Temperature Conversion Chart

Temperature°C	Digital Meter Response
16	.091 to .123
17	.100 to .135
18	.108 to .146
19	.118 to .159
20	.129 to .174
21	.138 to .187
22	.151 to .204
23	.164 to .222
24	.177 to .240

11.7 FUNCTIONAL TEST TROUBLESHOOTING

If you don't achieve good results with the functional test procedure, check the following:

Results	Solution
Too low	Be sure to inject the Hg vapor ONLY after the display flashes (2 seconds after SAMPLE is pressed).
Typically too high	Ensure the calibration vessel temperature is stable.
Too low	Ensure there is no oxidation on the mercury drop in the calibration vessel. Gently swirl the mercury drop in the calibration vessel. Replace if necessary.
Too low	Ensure the instrument's intake is not blocked with foreign matter. Check flow with a flow meter.
Too low	Use a new syringe needle. Straighten or replace crimped or blocked internal tubing.

If you find the above does not solve your problems, please call AZI Customer Service at (800) 235-3360 or 602-470-1414.

12 APPENDIX B - GOLD COIL PERSONAL MERCURY DOSIMETER

12.1 INTRODUCTION

The gold coil personal mercury dosimeter is a unique collection device for mercury vapor. The Jerome 431-X Gold Film Mercury Vapor Analyzer and the Personal Mercury Dosimeter determine personal exposure levels and ambient air concentrations, as well as low levels of mercury in natural and stack gases

For personal sample collection, the dosimeter is worn as close to the wearer's breathing zone as possible and is connected by tubing to a pump usually worn on a belt. The dosimeter can also be used for multiple point area monitoring by placing a dosimeter, with pump attached, in various strategic locations.

We recommend a pump flow rate of 2 cc/minute for the most accurate results when sampling in an atmosphere that for eight hours may contain an average of .5 mg/m^3 Hg. If you are considering using any other flow rate, see page 46, Nonstandard Flow Rates.

After sample collection is completed, the dosimeter is inserted in the Jerome 431-X's intake. A dosimeter lead set is connected between the dosimeter and the 25 pin connector on the back of specially equipped instruments. The instrument supplies power to volatilize the accumulated mercury from the dosimeter to the gold film sensor. The Jerome 431-X determines the mass of mercury collected by the dosimeter in a 17 second analysis. The dosimeter is ready for immediate re-use after a mercury measurement has been performed.

12.2 DOSIMETER TECHNICAL SPECIFICATIONS*

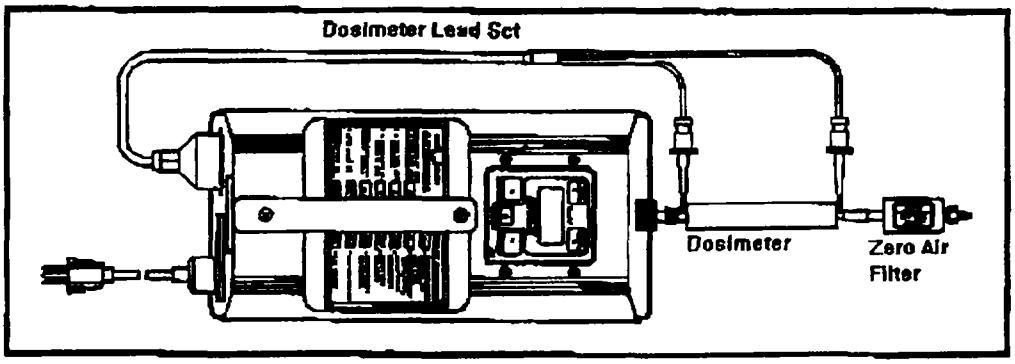
Sensitivity	< 0.5 x 10^{-9}g Hg
Precision	15% RSD @ 0.100 mg/m^3 Hg
Accuracy	15% @ 0.100 mg/m^3 Hg
Recommended flow rate	2 cc/min (0.002 liters/min) for atmospheres of 0.5 mg/m^3 60 cc/min for 100% collecting efficiency in atmospheres with lower concentrations
Construction	Nylon/Glass

Weight	1.5 ounces
Dimensions	0.5" dia. x 4.5" l
Capacity	1000 X 10^{-9}g Hg
Analysis Time	< 2 min

*Based on 2 cc/min flow rate

12.3 BEFORE SAMPLING WITH THE DOSIMETER

The personal mercury dosimeter adsorbs mercury vapor over a period of time. Therefore,



before each day's use it is necessary to ensure the dosimeter is mercury free. Perform the following steps to remove any accumulated mercury.

PROCEDURE:

! Connect the system as shown in the figure above.

Insert the dosimeter's large end in the 431-X's intake and gently tighten the intake tube nut to ensure an airtight seal.

! Attach the power cord to the 431-X and plug it into AC power.

AC power is required to heat the dosimeter.

! Press the Jerome 431-X's power ON button.

! Press the Jerome 431-X's SAMPLE button.

The digital meter reading will appear in 15 seconds.

! Wait 60 seconds and press the SAMPLE button again.

The Jerome 431-X's digital meter should display less than 0.005, verifying all mercury has been removed from the dosimeter coil.

! The dosimeter is ready for sample collection.

NOTE: For best results, dosimeter analysis should be performed as soon after collection as possible. If analysis cannot take place immediately after sampling is completed, cap both ends of the dosimeter with Tygon™ tubing sealing it completely, or replace with the end caps. For accurate results, perform dosimeter analysis no later than five days after sampling.

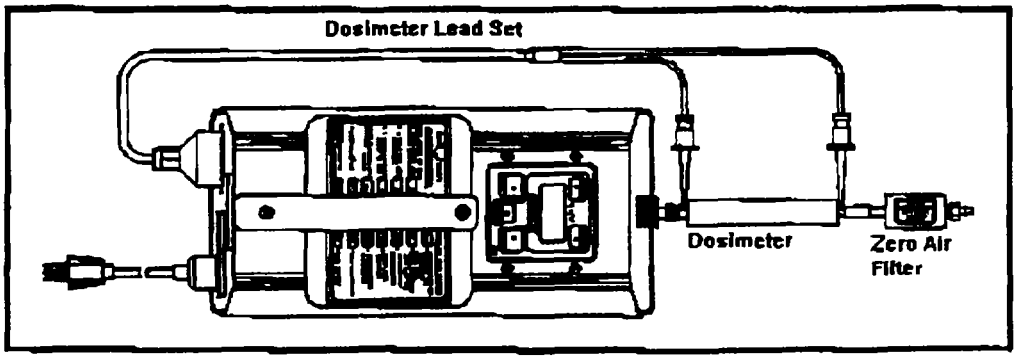
12.4 DOSIMETER ANALYSIS

PROCEDURE:

NOTE: Wait a minimum of 30 minutes after a sensor regeneration before starting this procedure.

! Connect the system as shown in the figure below.

! Attach the power cord to the 431-X and plug it into AC power.



AC power is required to desorb mercury from the dosimeter.

! Press the Jerome 431-X's power ON button and then press SAMPLE button.

The digital meter reading appears in 15 seconds.

! Record the digital meter reading (include the decimal point). Wait 30 seconds, then press SAMPLE again and record this digital meter reading.

Repeating the heating process ensures complete release of mercury from the dosimeter coil.

! Add the two digital meter readings together.

The sum of the two digital meter readings is the figure you will use in your calculations and is referred to as the meter response.

NOTE: A third dosimeter desorption should give .005 mg/m³ or less.

! You can perform the following calculation to obtain the mercury concentration in mg/m³ based on a time weighted average; **or alternately, DIP switch #2 can be set to OFF** and the digital meter will display nanograms Hg directly (refer to diagram, page 17).

Working Formula and Units of Measure
(MR x 87.5)/SV=Sample Concentration

MR(meter response)	total of the two digital meter readings in mg/m³
87.5 ng/mg/m³	(conversion factor, a constant which changes the meter response to nanograms of Hg)
SV (sample volume)	pump flow rate (in cc/min) multiplied by sample time (in minutes)
Sample concentration	in ng/cc mg/m³

EXAMPLE:
(to calculate a time weighted average during an 8 hour period)

Meter response	0.600 mg/m³ (sum of the two meter response readings)
Conversion factor	87.5 ng/mg/m³ (constant)
Pump flow rate	2 cc/min
Sampling time	8 hours (480 min)
Sample volume	2 cc/min x 480 min = 960cc

(0.600 mg/m³ x 87.5 ng/mg/m)/960cc = 0.055 ng/cc

A. Convert the meter response (the total of the two digital meter readings) to nanograms of mercury.

0.600 x 87.5 = 52.5 nanograms of Hg

B. Determine the total volume of air sampled.

2 cc/min x 60 min/hr x 8 hr = 960 cc

C. Determine the Hg concentration (time weighted average) of the dosimeter.
52.5 nanograms/960 cc = 0.055 ng/cc of Hg = 0.055 mg/m³ of Hg

! Check the sensor status after each dosimeter analysis.

IMPORTANT: Perform a sensor regeneration as soon as the meter display shows "----" (four bars) which shows 75-100% sensor saturation to prevent the loss of a sample.

! Seal the dosimeter with caps or Tygon™ tubing after analysis to prevent mercury contamination during storage.

NOTE: If your average dosimeter analysis produces nanogram levels of 75 or more, you risk overranging your instrument and losing your collection data. Call Customer Service at 800-235-3360 or 602-470-1414 for alternative collection methods.

12.5 Non-Standard Flow Rates and Dilution Modules

You may use a pump with a flow rate up to 50 or 60 cc/min, but be aware that there are certain limitations. If your pump flow rate exceeds 2 cc/min and your average dosimeter analysis produces nanogram levels of 75 or more, it may be easy to collect more mercury beyond the linear range of the 431-X sensor. You thus risk overranging your instrument and losing your collection data. Higher flow rates may also impair the capture efficiency of the dosimeter.

We recommend that you drop your flow rate or use a dilution module* (AZI P/N Z2600-3911). Lowering the flow rate to decrease the sample volume provides the greatest accuracy. Using a dilution module introduces an additional 15% inaccuracy to your analysis. As an alternative to the dilution module, sample for shorter time periods.

Dilution Module Specifications

Accuracy	+/- 15% of 10:1 ratio
Input concentration range:	
Low	0.7 mg/m³ Hg
High	5.0 mg/m³ Hg
Housing	Nylon
Dimensions	1" w x 2.7" l x 3" h
Weight	3.3 oz

The dilution module is factory set to a 10:1 ratio. The mass of mercury entering the dilution module is reduced by 90%, leaving a 10% (X10 dilution) concentration to be introduced into the Jerome 431-X. since this ratio can change slightly with use, it is important to occasionally determine the current dilution module ratio to ensure accurate

results. For normal applications a X8 to X12 ratio is recommended. The 431-X Functional Test Kit contains all accessories necessary to determine the current dilution module ratio.

Call Customer Service at 800-235-3360 or 602-470-1414 if you have questions about flow rates or applications.

**The dilution module contains Resisorb™, mercury vapor adsorbent. For safety information, see the Resisorb™ Material Safety Data Sheet on page 32.*

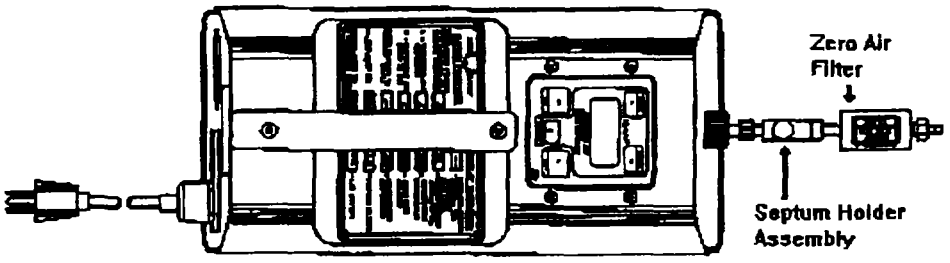
12.6 DILUTION MODULE RATIO CHECK

PROCEDURE:

NOTE: Wait a minimum of 30 minutes after a sensor regeneration before starting this procedure.

Direct 431-X Readings:

! Connect the instrument, septum holder assembly and zero air filter as shown in the figure below.



! Press the Jerome 431-X power ON button.

! Inject 1 cc of mercury saturated vapor into the septum, according to the Syringe Technique described on page 38 (431-X Functional Test, Appendix A).

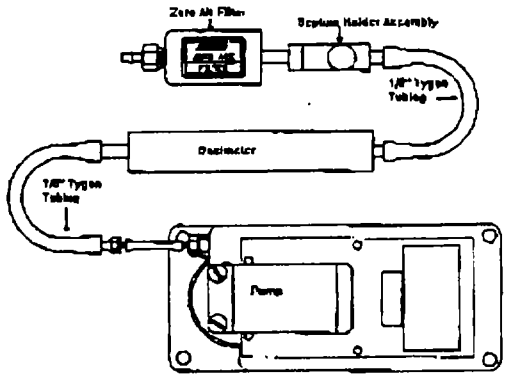
! Make 3 additional 1 cc injections and record the digital meter readings (include the decimal points).

! Average the results of the last 3 injections.

! Remove the septum assembly and zero air filter from the instrument.

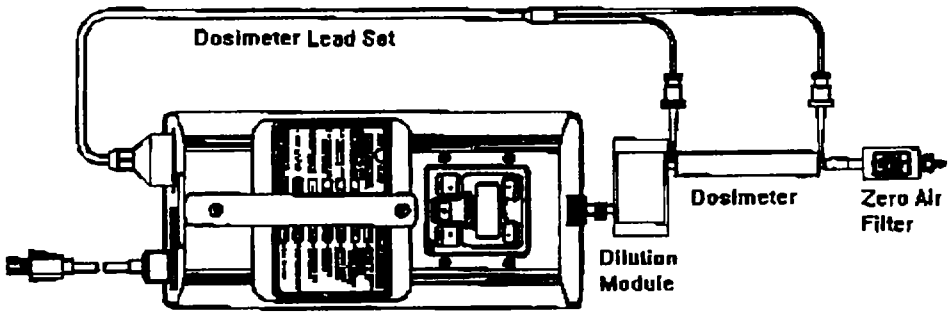
12.7 MOST ACCURATE METHOD

Perform the above test, however attach the dosimeter, septum holder assembly and zero air filter to the sampling pump that will be used. (See diagram.) Collection efficiencies should be approximately 100% up to 60 cc/min. If high flow rates are used, the final calculation should include this collection efficiency correction factor.



12.8 LOADING THE DOSIMETER

- Connect your pump, dosimeter, septum holder assembly and zero air filter (using 1/8" Tygon™ tubing) according to the figure at the right.
- Turn on the pump.



- Inject 1 cc of mercury vapor into the septum, ten times (total 10cc mercury vapor).
- Wait 30 seconds after the last injection, then turn off the pump.
- Remove the dosimeter, septum assembly and zero air filter from the pump.
- Connect the instrument, dilution module, dosimeter, zero air filter and dosimeter lead set as shown in the figure above.
- Attach the power cord to the 431-X and plug it into AC power.

AC power is required to heat the dosimeter.

- Press the Jerome 431-X power ON button and then press SAMPLE button.

The digital meter reading appears in 15 seconds.

! Record the digital meter reading (include decimal point). Wait 60 seconds, then press SAMPLE again and record this reading.

Repeating the heating process ensures complete release of mercury from the dosimeter coil.

! Add the two digital meter readings together.

The sum of the two digital meter readings is the figure you will use in your calculations and is referred to as the meter response.

! Repeat this procedcure two more times.

! Average the three meter responses you obtained in this section.

12.9 DILUTION MODULE RATIO CALCULATIONS

! Multiply the average obtained in the **Direct 431-X Readings** procedure by 10 (this is the number of 1 cc injections).

! Divide the result obtained in step 1 (above) by the average obtained in the **Loading the Dosimeter** procedure.

! Use the result as the dilution module ratio in your dosimeter analysis.

EXAMPLE:

Direct 431-X readings

0.102 mg/m^3
0.103 mg/m^3
0.104 mg/m^3
0.103 mg/m^3 average

Loading the dosimeter

0.120 mg/m^3
0.113 mg/m^3
0.100 mg/m^3
0.111 mg/m^3

Step 1 (above)
0.103 mg/m^3 x 10 = 1.030 mg/m^3

Step 2 (above)
(1.030 mg/m^3) / (0.111 mg/m^3 = 9.4

Dilution module ratio 9.4:1

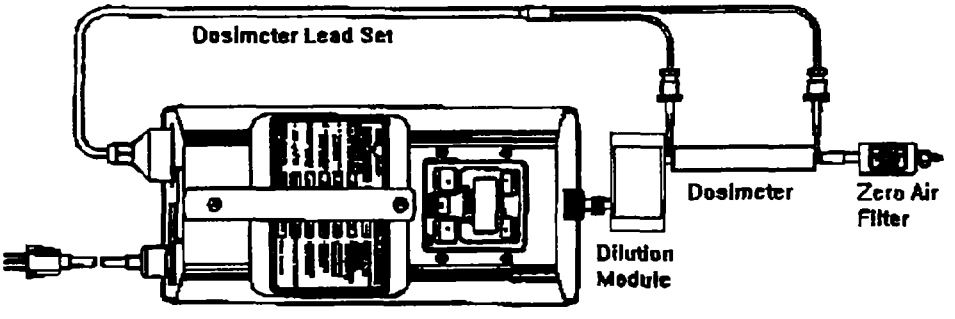
NOTE: For normal applications a X8 to X12 ratio is recommended. If your ratio is not within this range, call Customer Service at 800-235-3360 for assistance.

12.10 Analysis with a Dilution Module

PROCEDURE:

NOTE: Wait a minimum of 30 minutes after a sensor regeneration before starting this procedure.

- Connect the system as shown in the figure below.
- Attach the power cord to the 431-X and plug it into AC power.



AC power is required to heat the dosimeter.

- Press the Jerome 431-X power ON button and then press SAMPLE button. The digital meter reading appears in 12 seconds.

- Record the digital meter reading (include the decimal point). Wait 30 seconds, then press SAMPLE button again and record this reading.

Repeating the heating process ensures complete release of mercury from the dosimeter coil.

- Add the two digital meter readings together.

The sum of the two digital meter readings is the figure you will use in your calculations and is referred to as the meter response.

- *You can perform the following calculation to obtain the mercury concentration in mg/m³ based on a time weighted average.*

Working Formula and Units of Measure
Ng X DM/SV=Sample Concentration

Alternately, DIP switch #2 can be set to OFF and the digital meter will display nanograms Hg directly.

MR (meter response)	total of the two digital meter readings in mg/m³
87.5 ng/mg/m³.....	conversion factor, a constant which changes the meter response to nanograms of Hg
DM dilution module ratio.....	the ratio determined on page 50
SV (sample volume).....	pump flow rate (in cc/min) multiplied by sample time (in minutes)
Sample concentration.....	ng/cc = mg/m³

EXAMPLE:
(to calculate a time weighted average during an 8 hour period)

Meter response.....	0.600 mg/m³ (sum of the two meter response readings)
Conversion factor.....	87.5 ng/mg/m³ (constant)
Dilution module rate.....	9.4
Pump flow rate.....	2 cc/min
Sampling time.....	8 hours (480 min)
Sample volume.....	2 cc/min X 480 min = 960cc

(0.600 mg/m³ x 87.5 ng/mg/m)/960cc = 0.055 ng/cc

A. Convert the meter response (the total of the two digital meter readings) to nanograms of mercury.
The meter response multiplied by 87.5 (conversion factor) equals nanograms of mercury.
0.600 x 87.5 = 52.5 nanograms of Hg

B. Determine the actual mass of Hg collected by the dosimeter.
Nanograms of mercury times the dilution module ratio:
52.5 nanograms x 9.4 = 493.5 nanograms

C. Determine the total volume of air sampled.
The pump flow rate times 60 min/hr times 8 hours
2 cc/min x 60 min/hr x 8 hr = 960 cc

D. Determine the Hg concentration (time weighted average) of the dosimeter.
The mass of Hg collected by the dosimeter divided by the total volume of air sampled.
493.5 nanograms/960 cc = 0.0514 ng/cc of Hg = 0.0514 mg/m^3 of Hg

- Check the sensor status after each dosimeter analysis.

IMPORTANT: Perform a sensor regeneration as soon as the meter display shows "----" (four bars) to prevent the loss of sample.

- Seal the dosimeter with tubing after analysis to prevent excessive mercury contamination during storage.

Dosimeter Reference Chart 431-X

Expected concentration, related to sample volume and meter response

Volume of air in mg/m³ — **431-X meter response**

0.5	HL	HL	HL	HL	HL
0.1		0.549	HL	HL	HL
0.05		0.274	0.549	HL	HL
0.025	0.069		0.274	HL	HL
0.005	0.014	0.027		0.823	HL
0.001	0.003	0.005	0.011		0.329
	240	480	960	14,400	28,800

Volume of air in microgms/m³ (.001 micrograms/m³ = 1 nanogram/m³)

431-X meter response

0.5		0.494	0.823	HL	HL	HL	HL
0.05	0.025	0.049		0.274	0.823	HL	HL
0.01	0.005	0.010	0.016	0.055		0.329	0.494
0.005	0.002	0.005	0.008	0.027	0.082		0.247
0.0005	0.000	0.000	0.001	0.003	0.008	0.016	
	43,200	86,400	144,000	480,000	1,440,000	2,880,000	4,320,000

= Indicates the optimum meter response for that concentration, with the corresponding volume

Relationship of flow rate and time to total sample volume

Total volume collected (cc/minute)

1000	60,000	120,000	240,000	480,000	720,000	1,440,000	2,880,000	4,320,000
100	6,000	12,000	24,000	48,000	72,000	144,000	288,000	432,000
60	3,600	7,200	14,400	28,800	43,200	86,400	172,800	259,200
20	1,200	2,400	4,800	9,600	14,400	28,800	57,600	86,400
10	600	1,200	2,400	4,800	7,200	14,400	28,800	43,200
2	120	240	480	960	1,440	2,880	5,760	8,640
	1	2	4	8	12	24	48	72

Hours

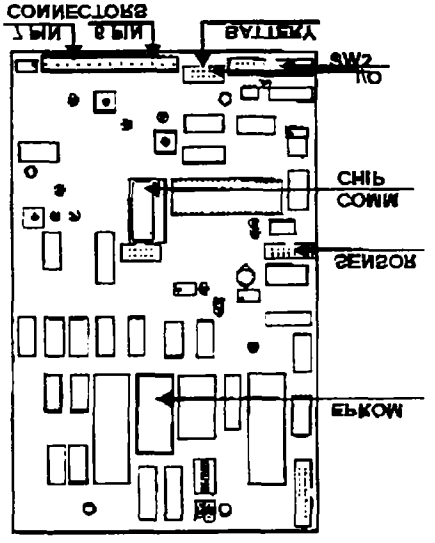
Use this formula for calculating the concentration of mercury in air:

Concentration (mg/m³) = Meter Response (x) 87.5 (a constant for the Jerome 431-X) / Flow Rate of sampling pump (x) Time

13 APPENDIX C - INTERNAL DIP SWITCH SETTINGS

Main (mother) board RED DIP switches (SW2)

This is the red DIP switch box located at the top, center of the instrument's main circuit board.



The 431-X provides regulated film heat at both 50 Hz and 60 Hz line frequencies. This also provides two ranges of preset but unregulated film heat (100-200/200-240 volt and 110-130/220-260 volt ranges). The preset film heats operate similarly to the old 431, but the two ranges are available to reduce the effects of chronic low or high line voltage.

Note: The ranges are doubled when the AC line selector switch is set to the 220V position. The DIP switch positions 1 and 6 must be properly set.

DIP 1	DIP 6	Function
OFF	OFF	60 Hz regulated film heat (100-130/200-260VAC)
OFF	ON	50 Hz regulated film heat (102-130/205-260 VAC)
ON	OFF	50/60 Hz preset film heat (110-130/220-260 VAC)
ON	ON	50/60 Hz preset film heat (100-120/200-240 VAC)

Regulated film heat should normally be used (DIP 1 OFF) except in the few cases where extremely dirty line voltage conditions may exist. These conditions might be found where large motors are being controlled or other situations may exist where the voltage may vary outside the 100-130 VAC range with regularity. In those cases the two preset heat ranges will allow some degree of satisfactory operation.

Switch Number	Normal Position	Action
2	ON	Nanograms mode
3	ON	Displays relative (not true) voltage during regen (0-255)
4	OFF	Display L-O-H when "zero" button pressed
	ON	Display 00-99 when "zero" button pressed
5	ON	Locks into 0-10mg/m^3 range (survey mode)

14 APPENDIX D - OPTION BOARD BLUE DIP SWITCHES

This is the blue DIP switch box located on the small (daughter) board mounted at the center of the main (mother) board.

Summary of blue DIP switch functions:

Switch	Function
1	Regeneration enable and time
2	Regeneration enable and time
3	Auto sample enable
4	Auto sample time
5	Auto sample time
6	DC power mode enabled (when ON and regeneration is started, this closes the relay on the data logger interface board to switch the inverter ON.

Timed Sensor Regeneration (Timed regeneration attempted one hour after start, then at interval.)

Switch #1	Switch #2	Regeneration Interval
Off	Off	Off
On	Off	6 hours
Off	On	24 hours
On	On	72 hours

Auto Sampling (Without JCI or data logger attached)

Switch #3	Switch #4	Switch #5	Sampling Frequency
On	On	On	No automatic sampling
Off	On	On	5 minutes
Off	Off	On	15 minutes
Off	On	Off	30 minutes
Off	Off	Off	1 hour

NOTE: Switch instrument power off before changing DIP switch settings.

15 APPENDIX E - OPTION BOARD MISCELLANEOUS TECHNICAL NOTES

15.1 INSTRUMENT ZEROING

The Jerome 431-X has essentially three zeros:

The instrument automatically rezeroes between samples and each sample is a unique reading. To take a sample, simply press the SAMPLE button.

The zero on the membrane switch is used to re-establish a baseline between the reference and sensor gold film after a sensor regeneration. This zero is manually adjusted by pressing the ZERO button and turning the potentiometer on the top of the instrument until the display reads 0. **Adjust only after sensor regeneration;** it is normal for H to be displayed after sampling.

The 431-X option board provides an auto zero feature that is invisible to the user. In some cases, the instrument does not resume sampling after a regeneration. At that time .L.L.L appears on the display when the ZERO button is pressed and the error message "manual bridge adjust needed" will be added to the notes column of the JCI text file if the JCI software is used. If this problem persists, it may be necessary to re-set the auto zero.

When necessary to re-adjust the auto zero point:

- Turn instrument off.
- Note original DIP switch settings.
- Turn DIP switch 4 on red DIP box to ON.
- Set the switches on the option board's blue DIP box to 1,2,6 OFF; 3,4,5 ON.
- Turn the instrument ON.
- Switch option board DIP #1 OFF and ON three times, leaving it ON.
- While pressing the ZERO button, turn the potentiometer on the option board until the numbers increase (maximum of 20). Note the display will flicker one digit.
- Return all switches to original position.

NOTE: The higher the auto zero number, the lower the capacity of the sensor and the more sensor regenerations are needed.

15.2 AUTOMATIC REGENERATION

The auto-regeneration should take place at the pre-configured time, with these exceptions: The instrument will always attempt a regeneration one hour after the SAMPLE button is pressed. The auto-regeneration will take place at the specified interval after that initial hour (for example, at hour 7, 13, 19, etc, if programmed for 6 hour auto-regeneration).

The instrument may NOT always perform the regeneration. The circuit forces the instrument towards 100% saturation to initiate the regeneration. If the sensor has not seen much mercury, the instrument will not auto-saturate. In this case, a regeneration will not take place. The instrument will always regenerate whenever the sensor is saturated. There should be no significant loss in sensitivity when a sensor auto-regeneration does not occur for 2-3 days.

Test this feature by initiating an auto-regeneration by turning the instrument ON and switching the daughter board's blue DIP switch #2 OFF and ON. If the line cord is plugged in, do not interrupt this regeneration cycle.

15.3 DC POWER MODE ENABLE

Instruments with the 431-X option board modification can be used with any +12 VDC source for continuous operation, if the AZI DC-AC power inverter kit is installed. The instrument requires 115 volts AC for regeneration. To preserve the life of the DC power source, the DC-AC inverter is switched on automatically for the regeneration only. The external switch on the inverter should always be OFF to preserve battery life during normal sampling.

When the instrument starts a regeneration and when DIP #6 is ON, the instrument sends a signal to close the relay on the 431-X data logger interface board mounted between the data logger and the instrument. This switches the inverter ON using the inverter's internal switch.

NOTE: When this mode is enabled, the instrument does NOT check for 115 VAC for the regeneration. If there is no AC power to the instrument, and a regeneration is initiated, the instrument will flash .H.H.H (rather than .P.P.P), however the sensor will not heat, nor will the sensor clean.

16 WARRANTY

Seller warrants to buyer that products delivered pursuant to this Agreement shall, at the time of delivery, and for a period of one (1) year thereafter (the Internal Battery Pack, where applicable, is warranteed for a period of ninety [90] days only), be free from defects in material or workmanship and shall conform to seller's specifications or such other specifications as seller has agreed to in writing. Seller's obligations with respect to claims under this warranty shall be limited, at seller's option, either to the replacement of defective or non-conforming product or to an appropriate credit for the purchase price thereof subject to the provisions of seller's Warranty Policy as amended from time to time, said Policy being incorporated herein by reference.

Return products under warranty claims will be shipped to seller's plant by buyer at buyer's expense and shall be accompanied by a statement of the reason for the return and an approved Return Material Authorization Number issued by seller. Buyer remains responsible for payment for products not accepted for warranty adjustment and freight and handling costs associated therewith.

Notwithstanding the foregoing, no warranty shall be enforceable in the event that product has been subjected to environmental or stress testing by buyer or any third party without written approval of seller prior to such testing. Further, no warranty shall be enforceable if the alleged defect is found to have occurred as a result of misuse, neglect, improper installation, repair, alteration, accident, or improper return handling procedure by buyer.

Discontinued product is warranteed only for a credit or replacement at seller's option.

THE EXPRESS WARRANTIES GRANTED ABOVE SHALL EXTEND DIRECTLY TO BUYER AND NOT TO BUYER'S CUSTOMERS, AGENTS, OR REPRESENTATIVES AND, EXCEPT FOR WARRANTY OF TITLE, IS IN LIEU OF ALL OTHER WARRANTIES, WHETHER EXPRESSED OR IMPLIED, INCLUDING ANY IMPLIED WARRANTIES OF FITNESS FOR A PARTICULAR PURPOSE AND MERCHANTABILITY, SUCH OTHER WARRANTIES BEING SPECIFICALLY DISCLAIMED BY SELLER. IN NO EVENT SHALL EITHER PARTY'S LIABILITY FOR ANY BREACH OR ALLEGED BREACH OF THIS AGREEMENT EXCEED THE TOTAL EXTENDED PRICE OR PRICES SHOWN ON UNFILLED ORDERS, NOR SHALL EITHER PARTY BE LIABLE FOR ANY SPECIAL, INCIDENTAL OR CONSEQUENTIAL DAMAGES RESULTING FROM BREACH OR ALLEGED BREACH.

Notwithstanding the foregoing, if any product covered by order(s) placed hereunder is designated as "developmental" "prototype" or "experimental," no warranty whatsoever except a warranty of title to component materials, will be applicable thereto and buyer shall indemnify seller for any claims for liability asserted seller in connection therewith.

Medical Applications: Seller's products are not designed for use in medical appliances, devices or systems where malfunction of buyer's product can result in personal injury. Buyer's customers using or selling buyer's products for use in medical applications do so at their own risk and agree to fully indemnify buyer.

The foregoing state the entire liability of seller in connection with products supplied hereunder.

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Arizona Instrument Corporation
Jerome 431-X Mercury Vapor Analyzer Operation Manual
Part Number SS-086
Revision C
July, 1996

If you have any questions regarding the operation of this instrument, please call our toll free number (800) 235-3360. Internationally, call (602) 470-1414 or fax (602) 470-1888.

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